September 20th, 2000

TECHNICAL RESEARCH REPORT
TO
IDRC

Transformation of Shea butter (Burkina Faso)
Final Report

PILOT PLANT CORP.

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Final Report

Species
Butyrospermum paradoxum
Butyrospermum parkii

Family
Sapotaceae

Herve Douce, Chem. Eng., Biochemist
Scientist, Oilseeds and Oils Processing

Approved for release by:

Paul Fedec, Ph. D.
Director of Quality Assurance
& Regulatory Compliance

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POS Project No. 56-97-856

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Study Personnel

Study Personnel	Job Title	Signature
Herve Douce, B.E, B.Sc.	Study Director	
Paul Orde, B. Sc.	Technician	
Gary Pocha	Technician	
Kin Ho, B. Sc.	Technician	
Myles Marianchuck, B.S.A.	Chief Analyst	
Luchun Wang, M. Sc.	Technician	

1. BACKGROUND

This report describes the objectives and work completed on IDRC Project File 02724; Transformation of Shea butter (Burkina Faso). The project was executed with the contracting agencies being POS Pilot Plant Corporation (POS) and the Department of Natural Substances of the Institut de Recherches en Sciences Appliquées et Technologie (IRSAT). Project initiation was January 1, 1998, with completion on September 15, 2000.

2. OBJECTIVES

Objectives listed are as proposed, defined and approved per IDRC file: 02724, Transformation of Shea butter (Burkina Faso).

The main objective of the current project is to improve the utilization of Shea butter through value added processing and quality improvements. The perceived dependence of Burkina Faso's economy on improved utilization of Shea demands an immediate, speedy and successful execution of this program. However, strategies have to be implemented now to ensure future sustainability and growth. The immediate, specific goals to attain are:

1. Evaluation and standardization of treatment methods to inactivate the inherent and contaminating microbial lipases in the Shea kernel.

2. Development and evaluation of refining and deodorization methods to improve the overall quality of locally produced Shea butter.

3. Exploration of possible alternatives to promote the domestic use of Shea butter.

4. Pilot plant scale-up of the developed or adapted technologies for Shea and production of product samples.

5. Determine the economic feasibility of developed or adapted technologies for use in Burkina Faso.

6. Training of one individual on laboratory methods and quality control. Training of one individual on management of an oils processing plant.

Objectives have been broken into phases, based on rough chronology of project execution at POS. Phase 4, 'Commissioning of RBD Technologies Lab Deodorizer' and Phase 5, 'Acquisition of De Smet Fractionation Unit' are included in the reporting but not covered under the primary objectives.

Objective 1. Evaluation of Harvest Techniques

Evaluation and standardization of treatment methods to inactivate the inherent and contaminating microbial lipases in the Shea kernel.

Status

Evaluation of harvesting, oil extraction techniques and analytical results has been executed by the Department of Natural Substances of the Institut de Recherches en Sciences Appliqués et Technologie (IRSAT). Results are reported in report entitled "Transformation du beurre de Karité, Project 02724, CRDI/IRSAT".

Objective 2. Evaluation of Refining Techniques

Development and evaluation of refining and deodorization methods to improve the overall quality of locally produced Shea butter.

Status

This objective has been broken down into three phases:

Phase 1A. Alkali Refining of Shea butter
Phase 1B. Physical Refining of Shea butter
Phase 1C. Solvent (in ethanol) Refining of Shea butter

This represents a deviation in detail but not in the primary objective, 'Development and evaluation of refining and deodorization methods to improve the overall quality of locally produced Shea butter'. See Results and Discussion section.

Objective - as per IDRC FILE: 02724

Two main refining processes are usually used in the fat and oil industry. Alkali refining involves neutralization of free fatty acids using sodium hydroxide followed by a bleaching step and finally a deodorization step. Physical refining involves a bleaching step followed by physical deodorization to remove the free fatty acids. These processes may be adaptable to Shea but cost and infrastructure constraints require that cost effective, and appropriate technologies be developed for the area. In this context the researchers are contemplating developing a solvent refining process to replace the conventional alkali refining process practiced in industries in Europe. The choice of a solvent refining process is based on the abundance of excess ethanol in Burkina Faso. The process envisaged would involve a simple stir tank agitation, time lagged phase separation and decantation of the refined butter from the solvent/contaminant mixture. The solvent is subsequently recovered for re-use while the fatty acids can serve as feedstock for the soap industry. The process is deemed to offer several advantages over the conventional alkali method with respect to Burkina's resources. The main advantages would be the anticipated lower energy consumption, hence lower cost and capital investment. The tasks to accomplish in the development of this novel refining process are:

a) Identification of the modulating factors; temperature, solvent/butter ratio, solvent composition, FFA content of butter, contact time and speed of agitation.
b) Optimization of the important modulating factors with respect to efficiency and yield.

c) Optimizing the solvent recovery process.
d) Process definition and specification.

The quality attributes that will be monitored during the refining process development are; FFA, moisture, colour, odour and non-saponifiables. Parallel refining comparison of the new method will be made with conventional alkali or physical refining method to compare efficiencies.

Bleaching tests would be conducted using activated charcoal and various clays. The quality factors that would be monitored in this test are; color, FFA and odor.

Deodorization of the butter has to be achieved without destroying the non saponifiables and other nutritionally important components of the butter. Factors such as; steam sparging rate, temperature, residence time and initial FFA will have to be optimized to achieve the goal of maximum retention of the important components. Quality attributes to monitor during the tests are: final FFA, odor, color and non-saponifiable matters.

Objective 3. Evaluation of Dry Fractionation

Development of a method to produce altered melting characteristics products for new markets.

Status

This objective has been broken down into two phases:

Phase 3A. Dry Fractionation of Shea butter
Phase 3B. Solvent Fractionation of Shea butter
Phase 3C. Coco butter substitute (CBS)

Objective - as per IDRC FILE: 02724

Additional technologies such as dry fractionation method (the separation of the solid phase from the liquid phase in a partly melted fat) will be explored with the view of enhancing and diversifying the utilization of Shea butter domestically or as an import substitution. Tasks to attain under this objective are:

a) Determine the appropriate dry fractionation conditions for Shea butter.

b) Separation of the solid (stearin) and liquid (olein) fractions from the butter.

c) Analysis of each fraction to evaluate its best possible use; it is possible that the highly priced cosmetic compounds might be enriched in one or another of the phase, depending on the operating parameters.

d) Development of prototype liquid cooking oil and margarine products from the appropriate fractions.

e) Analysis and tests of the sample products to verify compliance with quality requirements.

Objective 4. Scale Up

Scaling of bench-top processes to the pilot plant.

Status

This objective has been broken into two phases.

Phase 4A. Refining and fractionation of Shea butter in the Pilot Plant.
Phase 4B. Commissioning of RBD Technologies laboratory deodorizer.

About one tonne of crude Shea butter was air freighted from Burkina Faso to POS. The crude oil was bleached and physically refined.
Some of the refined oil was fractionated using hexane as carrier solvent. The olein and stearin fraction was fully desolventized and packaged.

Objective - as per IDRC FILE: 02724

In many cases, excellent technologies developed or successfully adapted in the laboratory become impossible or uneconomical to scale-up. It is, therefore, essential that the scalability of the developed or adapted technologies be tested in the pilot plant. It is also crucial that the stakeholders of this program; Burkina Faso people and their government, IDRC, associated NGOs and other organizations see tangible results of this program in terms of real products that can be tried. To meet this goal the investigators propose to conduct scale-up tests of the developed or adapted technologies at the POS Pilot Plant facility in Saskatoon, Canada. Half a tonne of crude Shea butter will be air freighted to Saskatoon for this purpose. Data on the scale-up tests would be used in subsequent economic evaluation of the processes. The refined or transformed products along with corresponding consumable products developed from them will be returned to Burkina Faso for presentation and testing by stakeholders.

Objective 5. Economic Feasibility

Assessment of economic feasibility.

Status

Phase 5: An economic feasibility analysis of the proposed refining and fractionation system was done after the scale up work.

Objective - as per IDRC FILE: 02724

Economic feasibility analysis based on input costs and potential revenues extrapolated from the laboratory and pilot plant scale-up results to industrial scale would be made. Based on the viability of the evaluated processes a proposed process flow for a demonstration pilot plant would be defined.

Objective 6. Training

Training of key personal in important aspects of oil processing and Shea butter processing

Status

This objective was altered and Mr. Kassamba Bakari was at POS for a single 8 week training period. This objective is reported as Phase 2.

Objective - as per IDRC FILE: 02724

Two training programs are proposed in collaboration with the Canadian partner:

1) Technical training in terms of laboratory methodologies and quality control determinations to support the pilot plant.

2) Training of a process engineer in hands-on management of a fats and oil processing pilot plant.

Both training programs are planned to be undertaken at the pilot plant processing facility of POS Pilot Plant Corporation in Saskatoon, Canada. Each training program will involve one individual and will be of four weeks duration.

IRSAT personnel will also be trained at the facilities of the De Smet company in Belgium, on the use of the equipment to be bought from that company.

Phase 1A. Alkali Refining of Shea butter

Summary

The Shea butter had a very strong tendency to emulsify with the water used for the alkali refining process. Water washing of the oil was not manageable and easily formed emulsions. Actual trial conditions are reported in Appendix 1A-1. This task has been aborted. It is believed that the high unsaponifiable content, 8.58%, is responsible for this oil's tendency to emulsify. This is one of the properties that make Shea butter suitable for cosmetic applications.

Analysis of the crude oil follows.

Table 1A-1. Crude Shea butter analysis

	Shea butter	Literature References [1]	Literature References [2]
Crude oil			
Peroxide Value, meq/kg	6.80		
p-Anisidine Value	3.40		
Free Fatty Acids, %	2.04	4.7-7.0	
Colour, 5.25" Lovibond (AOCS)	70.0Y2.9R		
Colour, 5.25" Lovibond (Lovibond)	70.0Y3.0R		
Gardner colour	5.2	9-10	
Chlorophyll, ppm	0.09		
Iodine Value	56.9	56.4-58.6	53-65
Unsaponifiables, %	8.58	5.6-7.6	2-11
Mettler Melting Point, °C	31.4		
Solid Fat Index, %			
10°C	39.8		
21.1°C	34.2		
26.7°C	20.7		
33.3°C	1.8		
40.0°C	0.3		

[1] Ensayos para la refinación fisica de la mantec de karité; Mendez, M.V., et al; Grasas y Aceites; Vol 42 (1991); pp 121-126

[2] The Lipid Handbook; Frank D. Gunstone et al, ed.; pub. Chaman & Hall, N.Y.; pp. 88-112

Table 1A-2. Crude Shea butter analysis - Minor Constituents

	Shea butter	Literature References [1]	Literature References [2]
Crude oil			
Phosphorus, ppm	<0.2	70-100	
Sulfur, ppm	<0.5		
Iron, ppm	1.48		
Copper, ppm	<0.05		
Fatty Acid Composition *, %			
C16	3.25	3.7-3.8	3.3
C18	43.86	38.3-37.5	44.3
C18:1	44.59	50.8-49.7	45.6
C18:2	5.92	6.5-7.9	5.5
C18:3	0.21	0.8-0.9	
C20	1.53		1.3
C20:1	0.35		
C22	0.14		
C24	0.08		
Tocopherols, µg/g	**	Traditional 780-902[3] Pressed 1720-1760	
Sterols, µg/g			
Delta 7 - Stigmastenol			944
Delta 7 - Avenastenol			281
Unknown			132

* Bleached using 0.2% phosphoric acid pretreat, 2.0% Supreme 120 FF clay at 110°C for 30 minutes, Deodorized at 250°C for 2 hours using 6% of steam/hr.
** Series of unknown compounds eluting at same time as sterols and tocopherols

Table 1A-3. Alkali Refined and bleached Shea butter oil

Conditions	Peroxide Value, meq/kg	Free Fatty Acids, %	AOCS 5.25' Colour R	AOCS 5.25' Colour Y	Gardner Colour
0.1% Citric Acid 15 minutes	0.54	0.47	0.7	9.1	1.8

[1] Ensayos para la refinación fisica de la mantec de karité; Mendez, M.V., et al; Grasas y Aceites; Vol 42 (1991); pp 121-126
[2] The Lipid Handbook; Frank D. Gunstone et al, ed.; pub. Chaman & Hall, N.Y.; pp. 88-112
[3] The level of tocopherol in Ghanaian Oils; J.K.B.A. Ata & Agnes Cobbina; Ghana Jnl agric. Sci., 6 (1973); pp 45-47

Phase 1B. Physical Refining of Shea butter

Summary

Phospholipid Removal

Initial protocols were established based on literature information that indicated a phosphorus content of 70-100 ppm (Table 1A-2). The phosphorus content of the oil received was <0.2 ppm. It is not know if the material received is representative of the long term quality of the Shea butter or if the specific processing used to generate this sample, resulted in the lower phosphorus content. If the low phosphorus content is related to the processing and the process is continued as per the sample received at POS then phosphorus content will not be an issue.

Bleaching

Initially single conditions were selected for bleaching to determine a starting point for oil quality (Table 1B-1). This was followed by a bleaching study using varying clay dosages (Table 1B-1) and several trials of atmospheric bleaching (Table 1B-2) to compare against vacuum bleaching. The bleaching clay used in the trials was Tonsil 'Supreme 120 FF'. There is an economic benefit if a vacuum system could be deleted from the system but it will impact oil quality. The protocol used for the trials is attached as Appendix 1B-1.

The minimum colour was obtained between 90°C and 100°C for the atmospheric bleached oil, Figure 1B-1. The vacuum bleached oil however had a lower bleached colour, Figure 1B-1. Clay dosages above 1.5% of Supreme 120 FF do not further reduce colour during vacuum bleaching, Figure 1B-2. The colour using 2% clay atmospheric bleaching was similar to the 1% clay vacuum bleaching, same Figure.

Atmospheric bleaching using Burkina Faso clay (2% at 100°C) yielded results comparable to Tonsil 120 FF clay, see Table 1B-2.

Deodorization (Physical Refining)

(Physical refining is the terminology used when free fatty acids are removed using steam stripping instead of alkali refining. The unit operation is the same as normal deodorization but more stripping steam capacity is required than for deodorization of alkali refining oil.)

Several of the bleached samples were lab deodorized as per the protocol in Appendix 1B-1. Results of the trials are listed in Table 1B-3. Peroxide values and para-anisidine values for the deodorized oils were not significantly different. Free fatty acids were reduced as the deodorization temperature increased from 0.15% at 220°C to 0.02% at 260°C using the same stripping time, 1 hour. The free fatty acid content of the lower temperature trials likely could have been reduced to the same free fatty acid level by increasing the deodorization time.

A comparison of the RBD colour is shown in Figure 1B-3. As can be seen the colour of the deodorized oil from the vacuum bleached oil is lower than the final colour from the atmospheric bleached oil, 0.5-0.6 Red compared to 0.7-0.9 Red. However in both cases the final colour compares favourably with commercially available RBD Shea that usually has a colour of 3 to 4 on the Gardner scale.
When atmospheric bleached oil is deodorized there is a tendency for the red component of the oil colour to increase during deodorization.

Table 1B-8 shows FAC and SFI were not significantly different from the crude to deodorized oil. There was a rise in unsaponifiable content, which may be a result of the removal of the free fatty acids, which may account for a 0.2% rise, and the rest may be variation in analysis. The AOM of 34.6 hours indicates a stable oil relative to many other vegetable oils. This is an expected result considering the oil has low poly-unsaturate content.

Table 1B-9 lists the FAC for the deodorized oil and deodorizer distillate. There is no significant difference indicating the free fatty acids have essentially the same fatty acid distribution as the triglyceride oil. This is expected for most oils.

Tocopherols and sterols content were established using a gas chromatograph operated according to established analytical protocols. A large number of unknown chromatographic peaks and over lap of various sterol peaks would not allow resolution and identification of the compounds. Since there is considerable literature information [3,4,5,6,7], on the sterols and tocopherol composition of Shea butter, identification was not pursued further.

Colour Scales

Tables 1B-4, 1B-5, 1B-6, and 1B-7 and Figures 1B-4 and 1B-5 show a comparison of colour data gathered from different colour measurement systems for the various trials. The data is provided for information purposes and future intent would be to be able to compare colour measurements produced or expected by third parties. The Lovibond and AOCS scale are very well correlated, $r^2 = 0.96 - 0.99$, Figure 1B-4. However the Gardner scale is not as well correlated to the AOCS red scale, $r^2 = 0.76$, Figure 1B-5. This result was expected since the AOCS and Lovibond scales are based on adsorption characteristics at the same wavelengths and the Gardner scale is set up using a different wavelength.

3: Sterols and methyl sterols in some tropical and subtropical vegetable oil; Toshihiro Itoh, Toshitake Tamura and Taro Matsumoto.

4: Characterization of Tripterpene alcohols of seed oils from some species of Theaceae, Phytolaccaceae and Sapotaceae; Toshihiro Itoh, Toshimitsu Uetsuki, Toshitake Tamura and Taro Matsumoto.

5: 24-Methylenelanost-9(11)-en-3 β-ol, New Tripterpene alcohol from Shea butter, T. Itoh, T. Tamura and T. Matsumoto.

6: The non-glyceride saponifiables of Shea butter; Kenneth E. Peers, J. Sci Fd Agric. 1977, 28, 1000-1009

7: The effect of processing on vegetable oil sterols , A literature review, S.P. Kochhar, M.Sc., Ph.D., A.I.F.S.T.

All trials using 2.0% clay.

Crude

Figure 1B-1. Effect of Bleaching Temperature on oil red colour

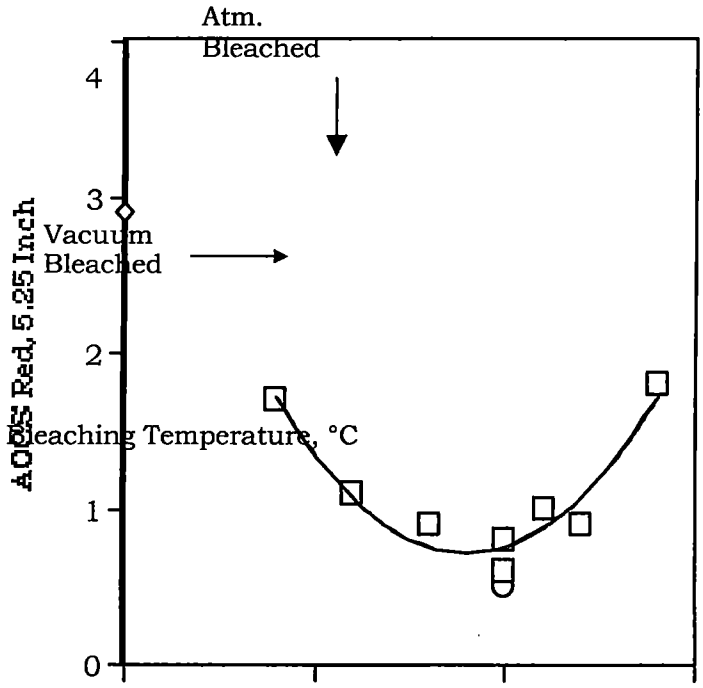


Figure 1B-2. Effect of Bleaching Clay Dosage on oil red colour

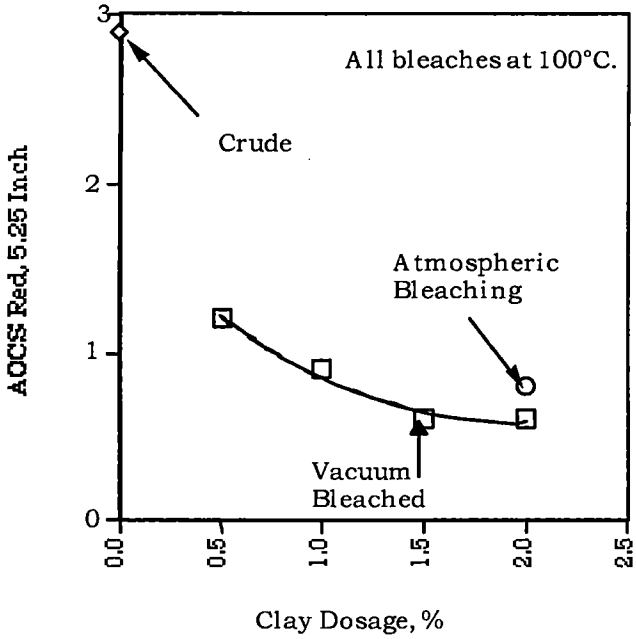


Figure 1B-3. Effect of Temperature on Deodorized oil red Colour

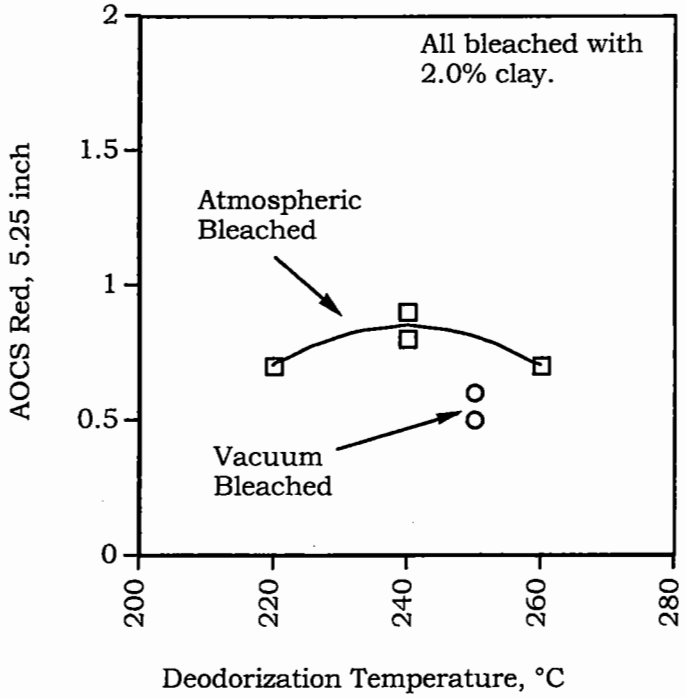
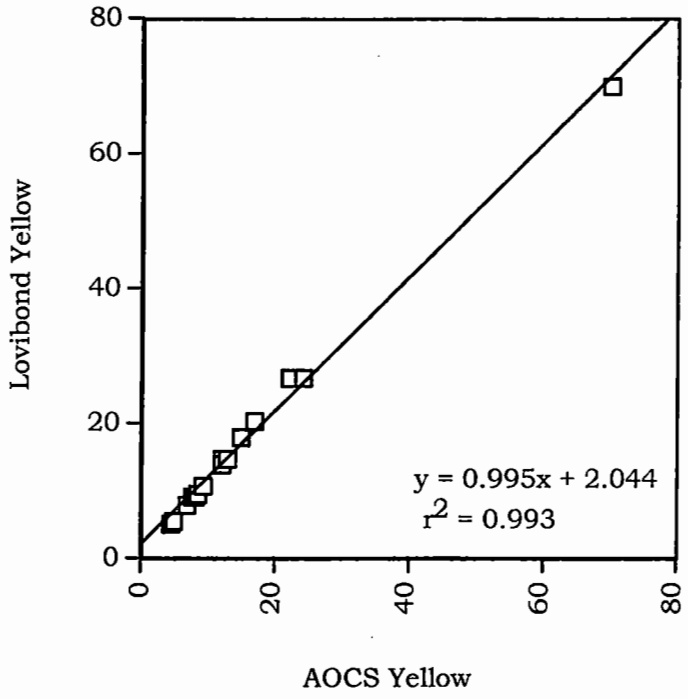
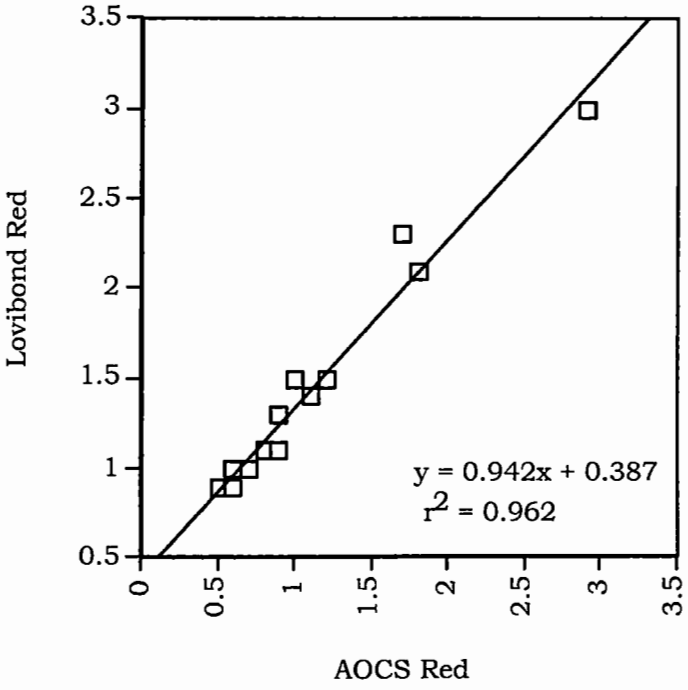


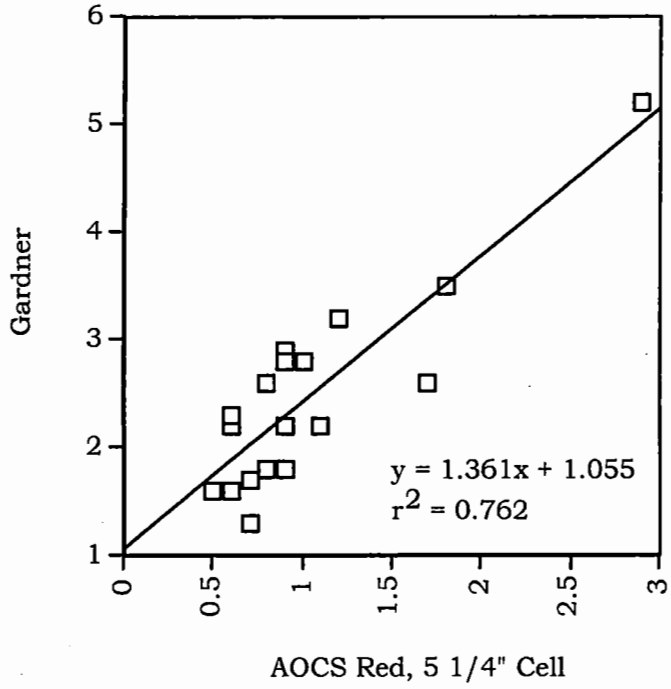
Figure 1B-4. Comparison of AOCS and Lovibond Colour Scales





Data for above two Figures from Table 1B-5.

Figure 1B-5. Comparison of AOCS Red and Gardner Scales



Data for above Figure from Table 1B-5.

Table 1B-1. Vacuum Bleaching Trials

Pretreatment as specified.
Oil charge: 1,100-1,200 g
Clay dosage: 2% Tonsil Supreme 120 FF unless otherwise specified.
Bleaching Temperature: 110°C
Bleaching Time: 30 minutes under vacuum
Filter Aid: 1% of oil weight

Conditions	Peroxide Value, meq/kg	Free Fatty Acids, %	AOCS 5.25' Colour		Gardner Colour
			R	Y	
0.1% Citric Acid, 15 minutes*	0.54	0.47	0.7	9.1	1.8
0.2% Citric Acid 15 minutes **	0.40	1.79	0.5	8.0	1.6
0.1% Citric Acid 15 minutes **	0.50	1.92	0.6	7.8	1.6
0.1% Citric Acid 15 minutes 0.5% Clay	-	-	1.2	24.0	3.2
0.1% Citric Acid 15 minutes 1.0% Clay	-	-	0.9	13.0	2.9
0.1% Citric Acid 15 minutes 1.5% Clay	-	-	0.6	9.4	2.2

* - Note soaps were 343.8 ppm and trial was aborted after bleaching.
**- Use in deodorization trials.

Table 1B-2. Atmospheric Bleaching Trials

No pretreatment
Oil charge: as specified
Clay dosage: 2% Tonsil Supreme 120 FF
Bleaching Temperature: as specified
Bleaching Time: 30 minutes, open container
Filter Aid: 1% of oil weight

Conditions	Peroxide Value, meq/kg	Free Fatty Acids, %	AOCS 5.25" Colour		Gardner Colour
			R	Y	
100g, Atmospheric at 40°C	2.93	-	1.7	17.0	2.6
100g, Atmospheric at 60°C	1.58	-	1.1	12.0	2.2
100g, Atmospheric at 80°C	0.92	-	0.9	12.0	2.2
100g, Atmospheric at 100°C	0.72	-	0.8	13.0	2.6
1604.5g, Atmospheric at 100°C *	**0.30**	**1.84**	**0.6**	**12.0**	**2.3**
489.5g, Atmospheric at 110°C	0.35	1.90	1.0	15.0	2.7
100g, Atmospheric at 120°C	0.60	-	0.9	15.0	2.8
100g, Atmospheric at 140°C	0.50	-	1.8	22.0	3.5
Atm. Bleach with 2% Burkina Faso clay at 100°C	**0.06**	**1.70**	**0.8**	**9.9**	-

* Used for deodorization trials

Table 1B-3. Deodorization Trials

Oil Type	Peroxide Value, meq/kg	p-Anisidine Value	Free Fatty Acids, %	AOCS 5.25" Colour		Gardner Colour
				R	Y	
Crude Shea butter	6.80	3.40	2.04	2.9	70.0	5.2
Vac. bleached, 0.1% pretreat	0.50		1.92	0.6	7.8	1.6
Deodorized at 250°C	0.12	-	0.01	0.6	4.8	<1
Vac. bleached, 0.2% pretreat	0.40		1.79	0.5	8.0	1.6
Deodorized at 250°C	0.00	2.71	0.02	0.5	4.6	<1
Atm. bleach at 100°C	0.30	-	1.84	0.6	12.0	2.3
Deodorized at:						
220	0.11	3.45	0.15	0.7	8.3	1.7
240*	0.00	3.16	0.14	0.8	8.4	1.8
240	0.00	3.43	0.07	0.9	8.6	1.8
260	0.00	3.24	0.02	0.7	6.9	1.3

All bleaches listed in this Table were executed using 2.0% of clay and a 30 minute bleaching time. The vacuum bleaching was done at 110°C and the atmospheric bleaching was done at 100°C .

* - Poor vacuum (3.7-8.0 mm Hg) occurred one hour after deodorization temperature was reached.

Note: Deodorization of vacuum bleached oil reduces the yellow component while leaving the red unchanged while deodorization of atmospheric bleached oil tends to increase the red component while reducing the yellow component. All deodorizations listed in the above Table were done in the glass deodorizer.

Note: Pressed, RBD **commercial** Shea has a colour specification of 3 to 4 on the Gardner scale.

Table 1B-4. Comparison of Colour Scales for Crude Shea butter

	AOCS Colour 5.25"				Lovibond Colour 5.25"				Gardner Colour
	Red	Yellow	Chl. a	Chl. b	Red	Yellow	Blue	Neutral	
Crude Shea butter	2.9	70.0	0.092	0.000	3.0	70.0	0.0	0.0	5.2

Table 1B-5. Comparison of Colour Scales for Vacuum bleached Samples

Standard conditions are 1100-1200g with 2% tonsil 120 FF clay at 110°C for 30 minutes under vacuum, filter with 1% filter aid.

Bleaching Conditions	AOCS 5.25" Colour				Lovibond 5.25" Colour				Gardner Colour
	Red	Yellow	Chl. a	Chl. b	Red	Yellow	Blue	Neutral	
Alkali Refined/bleached									
0.1% Citric Acid 15 minutes	0.7	9.1	0.000	0.000	-	-	-	-	1.8
Pretreat bleached									
0.2% Citric Acid 15 minutes	0.5	8.0	0.000	0.139	0.9	9.2	0.0	0.0	1.6
0.1% Citric Acid 15 minutes	0.6	7.8	0.000	0.000	0.9	9.2	0.0	0.1	1.6
0.1% Citric Acid 15 minutes 0.5% Clay	1.2	24.0	0.013	0.535	1.5	27.0	0.0	0.1	3.2
0.1% Citric Acid 15 minutes 1.0% Clay	0.9	13.0	0.010	0.228	1.1	15.0	0.0	0.1	2.9
0.1% Citric Acid 15 minutes 1.5% Clay	0.6	9.4	0.000	0.220	1.0	11.0	0.0	0.1	2.2

Table 1B-6. Comparison of Colour Scales for Atmospheric bleached Samples

Standard conditions are 1100-1200g with 2% tonsil 120 FF clay at 110°C for 30 minutes under vacuum, filter with 1% filter aid.

Bleaching Conditions	AOCS 5.25" Colour				Lovibond 5.25" Colour				Gardner Colour
	Red	Yellow	Chl. a	Chl. b	Red	Yellow	Blue	Neutral	
Atmospheric bleached									
100g at 40°C	1.7	17.0	0.003	0.230	2.3	20.7	0.7	0.0	2.6
100g at 60°C	1.1	12.0	0.000	0.108	1.4	15.0	0.0	0.0	2.2
100g at 80°C	0.9	12.0	0.000	0.038	1.1	14.0	0.0	0.1	2.2
100g at 100°C	0.8	13.0	0.000	0.000	1.1	15.0	0.0	0.2	2.6
1604.5g at 100°C	0.6	12.0	0.000	0.000	1.0	14.0	0.0	0.1	2.3
489.5g at 110°C	1.0	15.0	0.000	0.000	1.5	18.0	0.0	0.5	2.7
100g at 120°C	0.9	15.0	0.000	0.000	1.3	18.0	0.0	0.2	2.8
100g at 140°C	1.8	22.0	0.000	0.000	2.1	27.0	0.0	0.2	3.5

Table 1B-7. Comparison of Colour Scales for Deodorized oil

Deodorization Temp. (°C)	AOCS Colour 5.25"				Lovibond Colour 5.25"				Gardner Colour
	Red	Yellow	Chl. a	Chl. b	Red	Yellow	Blue	Neutral	
Vacuum bleached									
0.2% citric/deod. 250°C	0.5	4.6	0.000	0.035	0.9	5.2	0.0	0.1	<1
0.1% citric/deod. 250°C	0.6	4.8	0.000	0.000	1.0	5.6	0.0	0.0	<1
Atmospheric bleached									
220	0.7	8.3	0.000	0.000	1.0	9.5	0.0	0.1	1.7
240*	0.8	8.4	0.000	0.017	1.1	9.6	0.0	0.1	1.8
240	0.9	8.6	0.000	0.000	1.1	9.9	0.0	0.1	1.8
260	0.7	6.9	0.000	0.000	1.0	8.2	0.0	0.1	1.3

* - Poor vacuum (3.7-8.0 mm Hg) occurred one hour after deodorization temperature was reached.
Note: Pressed, RBD commercial Shea has a colour specification of 3 to 4 on the Gardner scale.

Table 1B-8. Miscellaneous Deodorized oil Analysis

	Shea butter **Crude**	Shea butter **Deodorized** *
Crude oil		
Unsaponifiables, %	8.58	10.09
AOM, hr	-	34.6
Solid Fat Index, %		
10°C	39.8	40.1
21.1°C	34.2	34.4
26.7°C	20.7	21.7
33.3°C	1.8	1.8
40.0°C	0.3	0.3
Fatty Acid Composition, %		
C16	3.25	3.24
C18	43.86	43.90
C18:1	44.59	44.61
C18:2	5.92	5.88
C18:3	0.21	0.19
C20	1.53	1.54
C20:1	0.35	0.35
C22	0.14	0.15
C24	0.08	0.08
Sterols & Tocopherols**		

* - Bleached using 0.2% phosphoric acid pre-treat, 2.0% Supreme 120 FF clay at 110°C for 30 minutes, Deodorized at 250°C for 2 hours using 6% of steam/hr.

**- Series of unknown compounds eluting at same time as sterols and tocopherols. Chromatograms have not been included.

Table 1B-9. FAC of Shea butter Distillate

	Shea butter **Deodorized** *	Shea butter **Distillate** **
Fatty Acid Composition, %		
C14	0.0	0.07
C16	3.24	5.40
C16:1	0.0	0.11
C18	43.90	47.42
C18:1	44.61	38.00
C18:2	5.88	6.11
C18:3	0.19	0.26
C20	1.54	1.31
C20:1	0.35	0.30
C22	0.15	0.12
C24	0.08	0.17

Note: Distillate and deodorized oil FAC 's were not from the same run.

* - Bleached using 0.2% phosphoric acid pre-treat, 2.0% Supreme 120 FF clay at 110°C for 30 minutes, Deodorized at 250°C for 2 hours using 6% of steam/hr.

** Bleached using 0.2% phosphoric acid pre-treat, 1.0% Supreme 120 FF clay at 110°C for 30 minutes, Deodorized at 250°C for 2 hours using 6% of steam/hr.

Phase 1C. Ethanol Refining of Shea butter

First Trial: (1 part ethanol: 4 part Shea butter)

A first attempt to disperse Shea butter in 95% ethanol followed by phase separation of the free fatty acids was not successful. The ethanol and Shea butter did not separate as distinct phases, rather an emulsion was formed. Trial protocol and observations are listed in Appendix 1C-1. This trial failed because an insufficient amount of ethanol was used to dissolve the Shea butter. (1 part ethanol : 4 part Shea butter)

Second Trial: (1 part ethanol: 0.17 part Shea butter)

A second set of trials was initiated in February 1999 using 95% by weight ethanol at a higher ratio of alcohol to Shea, (6 part by weight of ethanol: 1 part by weight of Shea butter). The first three trials of this set were done at 95°C while the last four were done at 120°C. The protocol is listed in Appendix 1C-2. Results are displayed in Tables 1C-1, 1C-2.

In these trials lowering the free fatty acid content of the starting bleached Shea oil was successful. The free fatty acid level dropped from an initial level of 1.65% in the starting bleached oil to a level of 0.34-0.25% in the bottom oil rich layer upon separation of Shea from ethanol. At the same time an enrichment of the upper oil poor layer with free fatty acid was observed, (1.65% to 7.32%).

Ethanol refining may be the most economic method for Burkina Faso in view of the abundance of excess ethanol and simplicity of the method.

Another advantage of the ethanol refining process is the possibility of latex removal from the Shea butter if so desired. The non polar latex is insoluble in ethanol. If the temperature of the Shea butter-ethanol mixture is chosen so that the Shea butter is completely miscible in the alcohol then the latex can be separated by filtration. At 120°C Shea butter is completely miscible in 95.6% by wt ethanol. Filtration at 120°C results in the separation of the latex from the Shea butter. However at 95°C Shea butter is not completely dissolved in the 95% by wt ethanol so the latex stays in the butter and little is recovered by filtration. See Table 1C-1.
The removal of the latex, ~ 1-1.5% by weight of the Shea butter, is required if a cocoa butter substitute is the end Shea product use, whereas if the end product is for cosmetic applications latex removal may not be required.

This method of latex and free fatty acid removal can be used with ethanol having a concentration of 85% by weight or higher, in a range of 1 to 24 parts by weight of alcohol to 1 part by weight of Shea butter[8]. The use of larger amounts of alcohol may not be economical. The upper, oil poor/alcohol layer obtained during separation can be repeatedly used for refining a new batch of Shea butter. However it is preferable to bleed off some of this layer and replace it with some fresh alcohol before reuse. The amount to be removed will depend on the initial free fatty acid content of the Shea butter but will usually be in the range of 5 to 20% by weight.

8: US patent 4, 103,039

Third Trial: (Refining using absolute ethanol)

See Table 1C-3 & 1C4 and protocol in Appendix 1C-3..

The main **disadvantages** of the refining trials using 95.6% by weight ethanol, (trial #2) are the requirement of a pressure vessel for reaching the temperature of 120°C and the requirement of pressure filtration at 120 °C. Both requirements would not be easily transferable to Burkina Faso.

A third refining trial using absolute ethanol was performed on the 24-25th of March 1999. Since Shea butter is more soluble in absolute ethanol the need for heating and filtering under pressure is eliminated.

Two trials were performed one using atmospheric bleached Shea and the other SM/Shea. More latex is removed during absolute alcohol refining than refining using 95.6% alcohol, i.e. (6.09 x 44.31%)/161.8=1.7% of latex removed when using absolute alcohol as compared to (1.67 x 45.12%)/125= 0.6% for the best trial using 95.6% alcohol.

The Shea-Absolute alcohol solution after filtration of the latex was then cooled to 10°C. This resulted in the precipitation of white Shea crystals, 61% by weight of the initial Shea dissolved in the alcohol. This fraction (S1) had a very low unsaponifiable level, 0.53%. The olein fraction, 39% by weight of the initial Shea dissolved in the alcohol, was enriched with the unsaponifiables, 11.41%. This method of fractionation of the stearin may represent the best method of obtaining a cocoa butter substitute, CBS, from Shea.

The main advantages of this method are:-

- The ability to refine the Shea stearin under low temperature (80°C). This will minimize energy requirement and any possible isomerization of the symmetrical triglycerides, SOS, POS, SOP which are needed for CBS.
- The possible fractionation of Shea in absolute ethanol excludes the need to use another solvent for that purpose.
- Ethanol is an abundant resource in Burkina Faso.
- The process does not need a pressure vessel or filtration under pressure.

The olein fraction obtained after desolventization yields a precipitate upon cooling to room temperature. This was filtered out to yield a second stearin fraction, S2 and an olein liquid fraction at room temperature. This olein fraction, 20% of initial Shea butter by weight, has a dropping point of 7.8°C and represents the best liquid Shea fraction obtained to date.

Table 1C-1: Latex precipitation and refining of bleached Shea butter using 95.6% by wt. of EtOH

Trial #1-#3
Mix 125g of bleached Shea & 750g EtOH, heat to 95°C
Hold 30 minutes
Filter through 1.0 micron glass micro fiber filter
Dry filter paper in 80°C oven for ~ 2 hr.
Phase separation of Shea from alcohol at 40°C

Trial #4-#7
Mix 125 g bleached Shea & 750g EtOH, heat to 120°C
Hold 30 minutes
Filter through 1.0 micron glass micro fiber filter (trial 4, 5, 7)
Filter through 0.45 micron Teflon filter for trial #6
Dry filter paper in 80°C oven for ~ 2 hr.
Phase separation of Shea from alcohol at 40 °C

	Trial #1	Trial #2	Trial #3	Trial #4	Trial #5	Trial #6	Trial #7
Temperature (°C)	95	95	95	120	120	120	120
Wt. of bleached Shea (g)	125	125	148.87	125	125	125	125
Wt. of 95.6% ethanol (g)	750	750	893.2	750	750	750	750
Wt of alcohol rich top layer (g)	-	726.38	859.3	717.55	735.63	744.31	762.85
Wt. of oil rich bottom layer (g)	-	114.03	132.2	111.39	103.87	88.73	84.95
% by wt of water in alcohol rich layer	-	4.03	3.99	-	-	-	-
% by wt of water in oil rich lower layer	-	-	0.31	-	-	-	-
Wt of oil obtained from alcohol rich layer (g)	19.52	25.58	24.70	20.19	29.53	39.08	48.18
Wt. of oil obtained from oil rich layer(g)	94.44	96.74	114.22	99.47	91.57	79.25	74.03
% oil in alcoholic top layer [b]	-	3.52	2.87	2.81	4.01	5.25	6.3
Alcohol Insoluble (g)	0.4865*	0.5100*	0.5061*	1.1168**	1.46**	1.67**	0.93**

* Residual on filter paper, very little or no latex (reaction and filtration done at 95°C)
** Latex was obtained on filter paper when reaction and filtration done at 120°C.
[b] $\frac{\text{Wt. of oil obtained from alcohol rich layer}}{\text{Wt of alcohol rich top layer}}$

Table 1C-2 : Analytical data for latex removal and 95.6% ethanol refining

Sample ID	PV meq/kg	Colour Y	Colour R	% FFA	Unsaponifiable %	Sap. Value	M.P. °C	Moisture %
SM/Shea #2	8.30	40	2.6	1.70	6.13	178.7	30.8	-
Bleached oil (Atm. 100°C)	0.12	9.1	0.7	1.65	5.91	179.3	-	-
Ethanol Refining								
Latex †	-		-	-	-	-	-	-
Oil from upper EtOH layer	-		-	7.32	-		-	3.99
Oil from lower EtOH layer	-		-	0.34	5.65		-	0.31
Latex ©	-		-	-	45.12			-
Oil from upper EtOH layer ©				-	8.30*/7.64**			-
Oil from lower EtOH layer©	-			0.25	4.68*/5.94**			-

† : Pooled sample of trial #1 to 3, filtration performed at 95°C .
©: Pooled sample of trial 4 to 7, filtration performed at 120°C .
* Better separation of lower oil rich layer from upper alcohol rich layer for trial #4&5
** Bad separation of lower oil rich layer from upper alcohol rich layer for trial #6&7

Table 1C-3: Latex precipitation and refining of bleached Shea butter using absolute EtOH

Trial #1

Mix 161.8 g of bleached Shea & 3,883g absolute EtOH.
Heat to 80°C, hold 120 minutes
Decant off alcohol-Shea solution at 80°C
to separate latex from solution.
Cool down solution to 10°C, Hold 1 hour.
Oven-dry crystals overnight. (S1)
Desolventize the alcohol/Shea fraction (O1)
Allow O1 to stand at room temperature for 24 hr
Decant liquid part of O1, O2 from precipitated crystals S2.

Trial #2

Mix 124.7g of **SM/Shea** & 3,883g absolute EtOH.
Heat to 80°C, hold 120 minutes
Decant off alcohol-Shea solution at 80 °C
to separate latex from solution.
Cool down solution to **15°C**, Hold 1 hour.
Oven-dry crystals overnight. (S1)
Desolventize the alcohol/Shea fraction (O1)
Allow O1 to stand at room temperature for 24 hr
Decant liquid part of O1, O2 from precipitated crystals S2.

	Trial #1	Trial #2
Temperature (°C)	80	80
Wt. of bleached Shea (g)	161.8	124.7
Wt. of absolute ethanol (g)	3,883.2	2,992.8
Alcohol Insoluble (g), mainly latex % wt of alcohol insoluble	6.09 3.8	2.69 2.2
Fractionation temperature, °C	10.0	15.0
Wt. of dried crystals after hold (g) (S1)	86.5	56.4
% wt of Stearin fraction, S1, e.g. 86.5/(86.5+54.9)	61.2	47.5
Wt. of Shea obtained from alcohol layer after hold (g) (O1)	54.9	62.3
% wt of O1	38.8	52.5
Wt. of liquid oil obtained from O1, i.e. O2 at 25°C(g)	28.52	-
Wt. of crystals obtained from O1, i.e. S2 at 25°C(g)	17.65	-

Table 1C-4: Analytical data for latex removal & Absolute ethanol refining

Sample ID	PV meq/kg	Colour Y	Colour R	% FFA	% Unsap.	Sap. Value	Iodine value	Dropping Point °C
SM/Shea #2	8.30	40	2.6	1.70	6.13	178.7	-	30.8
Bleached oil (Atm. 100°C)	0.12	9.1	0.7	1.65	5.91	179.3	56-58	-
Abs. Ethanol Refining #1 (Using Bleached Shea #2)								
Latex	-		-	-	44.31	-	-	-
S1	-	2.5	0.5*	0.05	0.53		39.14	34.9
O1	-		-	-	11.41		-	-
O2		3.3	0.5*		11.32		72.23	7.8
S2		3.4	0.6*		11.56**		67.95	36.4
Abs. Ethanol Refining #2 Using SM/Shea								
Latex	-		-	-	81.04	-		-
S1	1.92	1.8	0.5*	0.18	1.83		35.61	36.5
01	13.60	8.5	0.6*	3.10	9.86		68.02	18.1
O2					-		52.23	
S2					-		50.02	

* 1" cell AOCS Lovibond otherwise 5.25" cell.
** Calculated

Phase 2. Training

Summary

Mr. Kassamba Bakari was at POS from May 25, 1998 to August 24, 1998 for training on process methods used for the Shea butter and for training on analytical methods. Some of the training time was also used to determine process characteristics of the Shea butter.

Process training included physical refining, bleaching studies, and deodorization studies.

Bench top Procedures training included:

Degumming
Refining
Bleaching
Deodorization
Fractionation

Analytical Procedures training included:

Peroxide Value
p-Anisidine Value
Free Fatty Acids
Soaps
Iodine Value
Unsaponifiables
Solid Fat Index
Mettler Dropping Point
Tintometer Colour Reading (both digital and visual)
Fatty Acid Composition (observed principles only)
Tocopherols & Sterols (observed principles only)
Metals Analysis (observed principles only)

Phase 3A. Dry Fractionation

Summary

Satisfactory liquid and solid separation during the dry fractionation process was not achieved. Several trials were executed and only a single trial using a separation temperature of 31°C was successful and only produced a 5% stearin cut. The olein fraction was re crystallized at 29°C but the stearin and olein formed a gelled mass, and could not be separated. Some of the trial conditions are listed in Appendix 3A-1, and abbreviated results listed in Table 3A-1.

A blend of 50% by weight of Shea butter with 50% by weight of sunflower oil, an oil that does not solidify at room temperature, was used in another series of dry fractionation trials. Good fractionation yield was obtained at 28.3°C . The cut was 17.8% by weight stearin and 82.2% by weight olein(Table 3A-1). Judging from the stearin and olein yields the Shea butter was fractionated into a 35/65 split, i.e. 35% of initial Shea weight was in the stearin fraction and 65% of the original Shea butter was in the Olein fraction. This is supported by the distribution of the triglycerides in the two fractions as shown in Table 3A-2. SOS, the major triglyceride of Shea, was preferentially found in the stearin while SOO, the second most abundant triglyceride of Shea, was preferentially found in the olein fraction. About 88% of the initial SOO present in the Shea was preferentially found in the olein fraction while about 43% of initial SOS in Shea butter was found in the stearin fraction. To increase the efficiency of separation of SOS from SOO one may lower the fractionation temperature from 28.3°C to a temperature above 25°C,(Table 3A-1).
The unsaponifiables distributed themselves equally in the stearin and olein fraction. Using the 50/50 blend may allow for a liquid Shea/sunflower oil at room temperature, the olein having a dropping point of 22.5°C . The stearin fraction with a dropping point of 36.6°C could be used in the cosmetic industry in view of the high unsaponifiable content. Decreasing the fractionation temperature as suggested above will decrease the dropping point of the resulting stearin & olein fraction.

The fractionation process may need to be executed in solvent. This will be especially true if a stearin fraction with low unsaponifiable content is required, however this is a major constraint from a commercial standpoint as extra costs are involved in a solvent fractionation process.

The DeSmet fractionation unit obtained for IRSAT in Burkina Faso may be able to produce better results than those obtained on POS equipment.

Table 3A-1: Dry fractionation Trials

Date of trial	Type of Shea butter used	Temp at hold °C	Hold Time hr	Stearin fraction %	Olein fraction %	Comments
A: Crude Shea butter						
6/12/98	SM/Shea	24.6	1.0	~100	-	Stearin solidify in funnel
6/15/98	SM/Shea	28.0	2.0	-	~100	Very small crystals, poor filtration
B: Bleached Shea						
6/17/98	bleached Shea	22.5	0.5	~100	-	Excessive crystal formation
6/18/98	bleached Shea	26.4	16.5	~100	-	Complete crystallization
8/28/98	bleached Shea	29.0	16.0	~100	-	Complete crystallization
9/1/98	bleached Shea	30.0	20.0			Many crystals, could not separate by centrifuge
8/27/98	bleached Shea	31.0	20.5	5.66	94.34	Stearin D.P 35.8°C, Olein D.P 30.5°C
9/2/98	Olein fraction from 8/27/98	29.0	19.0			Semi solid mass, could not filter , will need high pressure filtration.
9/3/98	Olein fraction from 8/27/98	31.0	20.0	-	100%	No crystals present
C: Bleached Shea/RBD Sunflower blend, 50:50 by wt blend						
12/14/98	50/50 Shea/Sun	18.5	0.75			Complete crystallization
9/22/98	50/50 Shea/Sun	22.0	48.0			Complete crystallization
12/10/98	50/50 Shea/Sun	25.0	16.0	45.32 DP: 31.5°C	50.58 DP: 13.5°C	This suggests that most of the Shea has crystallized out.
12/15/98	50/50 Shea/Sun	28.3	20.0	17.8	82.2	Stearin D.P. 36.6 °C, Olein D.P. 22.5°C
D: Bleached & EtOH Refined Shea butter						
3/12/99	EtOH Bl. Shea	23	48.0	~100%		Complete crystallization
3/15/99	EtOH BL. Shea	30.6	20.0	~100%		Complete crystallization
3/16/99	EtOH Bl. Shea	31.0	4.0	~100%		Complete crystallization
3/17/99	EtOH Bl. Shea	34.3	19.5	~100%		Complete crystallization
3/19/99	EtOH Bl. Shea	36.6	48.0		~100%	No crystallization
3/23/99	EtOH Bl. Shea	35.2	20.0	~20%	~80%	Some crystallization

Table 3A-2: Dry fractionation of 50/50 Shea butter and Sunflower blend.

Triglyceride	% in Shea butter	% in Sunflower	% in Stearin from 50/50 Shea/Sun 35% split	% in Olein in from 50/50 Shea/Sun 65% split
LLL	-	34.20	8.70	21.79
LnLO	-	2.53	0.61	1.14
LLO	-	27.83	6.39	15.66
LLP	-	10.51	2.21	4.91
LOO	0.51	7.07	1.87	4.08
LLS	0.50	7.36	1.69	4.19
LOP	0.44	4.06	0.96	2.55
LPP	-	0.37	-	-
OOO	3.64	2.30	1.92	3.81
LOS	3.57	1.86	2.06	3.38
POO	1.28	0.54	1.05	1.21
SLP	0.60	0.62	0.83	0.84
SOO	32.64	0.26	7.88	17.45
SSL	3.65	-	2.21	1.87
SOP	**3.14**	-	**2.35**	1.45
POP	**0.50**	0.51	-	-
SOS	**46.60**	-	**57.02**	14.59
SPS	0.96	-	1.36	-
SSS			0.89	-
Total	98.03	100.02	100	103.4

P: Palmitic
S: Stearic
O: Oleic
L : Linoleic
Ln: Linolenic
Bolded triglycerides are the major symmetrical TG found in cocoa butter.

Phase 3B. Solvent Fractionation

Summary

Several trials were conducted and separations obtained. Results obtained from the various fractions are listed in Tables 3B-1, 3B-2 & 3B-3.
Please note that the triglyceride SOS is nearly exclusively fractionated in the stearin fraction while SOO is mainly found in the olein fraction. Extending the hold time or lowering the temperature during the hold may render the separation of SOS from SOO more complete.

Unsaponifiables
See Table 3B-4.
For dry fractionation the unsaponifiables biased towards the stearin fraction and for solvent fractionation the unsaponifiables biased towards the olein fraction. In general, high unsaponifiables will be desired for cosmetic applications (although functional thresholds are not known) and low unsaponifiables desired for food applications. Using this argument solvent fractionation would be the desired process for the cocoa butter substitutes (CBS). The concentration of unsaponifiables in the olein fraction from the solvent process with higher unsaponifiable content may have better functional properties for cosmetics than the non-fractionated oil.

It may be the presence of the unsaponifiable material and its emulsion properties that hinder the separation of olein and stearin in the dry fractionation process. The dry fractionation process however still has a distinctive processing cost advantage over solvent fractionation and the process ideally needs to be driven towards dry fractionation for economic reasons.

Most of the unsaponifiables of Shea butter have a boiling point of 240-270°C at 1 mm of Hg. As deodorization temperature is increased from 220°C to 260°C the amount of unsaponifiables in the distillate increased as would be expected, see Table 3B-4.
The amount of unsaponifiables lost during deodorization is a function of the temperature used and the time period the oil is at temperature. As can be seen from the residual unsaponifiables in the deodorized oils, the actual loss at the temperature investigated is minimal. To minimize unsaponifiables losses one could deodorize at temperatures approaching 200-220°C. This will also minimize any possible isomerization of the triglycerides specially the symmetrical triglycerides, SOS, POS & POP needed for cocoa butter substitutes.
If the aim is to reduce the unsaponifiable content then deodorization at 270°C , (two passes) will most likely remove a substantial amount of unsaponifiables.

Table 3B-1. Solvent Fractionation Analytical Data

ST- Stearin; OL - Olein

Date of trial	Fractionation temperature [a]	Shea : Hexane Ratio (by wt)	AOCS Colour (5.25")		Chlorophyll		Iodine Value	Dropping Point (°C)	Stearin, % Yield [b]
			Red	Yellow	a	b			
7/10/98	6°C ST [c]	25:75	-	-	-	-	33.42	40.2	21.14
7/10/98	6°C OL [c]	25:75	-	-	-	-	66.62	27.3	-
7/10/98	5°C ST [d]	25:75	2.6	12.0	0.110	0.157	40.48	38.9	34.41
7/10/98	5°C OL [d]	25:75	0.5	14.0	0.000	0.299	66.38	21.9	-
7/13/98	5°C ST	25:75	2.6	12.0	0.100	0.119	37.51	40.3	34.08
7/13/98	5°C OL	25:75	1.1	17.0	0.000	0.257	64.23	19.3	-
7/16/98	5°C ST	25:75	4.2	20.0	0.000	0.000	40.38	40.3	30.82
7/16/98	5°C OL	25:75	0.5	15.0	0.000	0.272	63.92	24.0	-
7/14/98	5°C ST	50:50	1.4	11.0	0.010	0.205	46.26	36.0	52.88
7/14/98	5°C OL	50:50	0.6	19.0	0.010	0.341	68.21	12.8	-

[a] Produced by heating bleached Shea butter (produced on 6/16/98) and hexane to 50°C then cooling to fractionation temperature. The cooling rate was approximately 0.34°C/minute, agitation was set at 30 RPM, and the holding time at temperature was 24 hours. Olein and Stearin were separated by filtration through Whatman #5 filter paper.

[b] The % Yield was calculated by the following formula: 1- ((olein (g)/ initial mass of Shea butter) X 100)

[c] This fraction was produced with no agitation.

[d] This fraction was held at 15°C for 21 hours and then cooled to 5°C and held there for 24 hours.

The trials on 7/13/98 and 7/16/98 are duplicates.

Note: All stearin looked cloudy in the 5.25" path length

Table 3B-2. Solvent Fractionation Color Data

ST- Stearin; OL - Olein

Date of trial	Fractionation temperature [a]	Shea : Hexane Ratio (by wt)	AOCS Colour (5.25")				Lovibond Colour (5.25")				Gardner Colour
			Red	Yellow	Chl. a	Chl. b	Red	Yellow	Blue	Neutral	
7/10/98	6°C ST [b]	25:75	-	-	-	-	-	-	-	-	-
7/10/98	6°C OL [b]	25:75	-	-	-	-	-	-	-	-	-
7/10/98	5°C ST [c]	25:75	2.6	12.0	0.110	0.157	3.3	16.1	1.1	0.0	2.1
7/10/98	5°C OL [c]	25:75	0.5	14.0	0.000	0.299	1.0	16.0	0.0	0.2	2.6
7/13/98	5°C ST	25:75	2.6	12.0	0.100	0.119	3.7	15.8	1.8	0.0	2.0
7/13/98	5°C OL	25:75	1.1	17.0	0.000	0.257	1.4	20.0	0.0	0.1	2.7
7/16/98	5°C ST	25:75	4.2	20.0	0.000	0.000	6.0	29.5	3.5	0.0	2.9
7/16/98	5°C OL	25:75	0.5	15.0	0.000	0.272	1.0	18.0	0.0	0.3	2.7
7/14/98	5°C ST	50:50	1.4	11.0	0.010	0.205	1.8	13.0	0.0	0.0	2.0
7/14/98	5°C OL	50:50	0.6	19.0	0.010	0.341	1.1	21.0	0.0	0.4	3.0

[a] Produced by heating bleached Shea butter (produced on 6/16/98) and hexane to 50°C then cooling to fractionation temperature. The cooling rate was approximately 0.34°C/minute, agitation was set at 30 RPM, and the holding time at temperature was 24 hours. Olein and Stearin were separated by filtration through Whatman #5 filter paper.
[b] This fraction was produced with no agitation.
[c] This fraction was held at 15°C for 21 hours and then cooled to 5°C and held there for 24 hours.

Note: All stearin looked cloudy in the 5.25" path length

Table 3B-3: Wet fractionation of 25/75 Shea butter and hexane at 5°C .

Fractionation done on the 7/13/98

Triglyceride	% in Shea butter	Stearin from 25/75 Shea/hexane	Olein from 25/75 Shea/hexane
?	0.26	-	0.54
LnLnLn ?	0.50	-	0.79
LaLaLa ?	0.43	-	0.78
LaLaL ?	0.78	-	1.27
LOO	0.51	-	0.94
LLS	0.50	-	0.63
LOP	0.44	-	0.69
OOO	3.64	-	6.17
LOS	3.57	-	6.08
POO	1.28	-	2.32
SLP	0.60	-	1.11
SOO	32.64	1.70	54.79
SSL	3.65	2.30	4.45
SOP	3.14	3.65	2.66
POP ?	0.50	-	0.90
SOS	**46.60**	**89.21**	15.06
SPS	0.96	2.01	0.80
SSS	-	1.13	
Total	98.03		

La: Lauric acid
?: Identity of triglyceride uncertain.
Please note the fractionation of the triglycerides results in SOS being fractionated mainly into the stearin phase & of SOO into the olein phase.
Bolded triglycerides are the major symmetrical TG found in cocoa butter.

Table 3B-4. Unsaponifiable Material

S: Steam PR: Physically refined	Unsaponifiables AOCS Ca 6b-53 Marine oils* %	Unsaponifiables AOCS Ca 6a-40 Non-Marine oil %
Crude Shea butter #1 (5/29/98)	4.98	8.58**
Crude Shea butter #2 (summer 98)	6.13Ψ	-
Physically Refined Shea butter, 250°C /2 hr., 6% steam,(S)	5.63	10.09 **
220°C /3 hr./6%S Distillate 2/4/98 (Shea #2)	1.42	-
220°C /3 hr./6%S 2/4/98 (PR Shea #2)	5.39	-
250°C /2 hr./6%S Distillate 6/19/98	9.43	-
250°C /2 hr./6%S 6/19/98(PR Shea)	5.42	-
260°C /2 hr./6%S Distillate 1/8/99	11.31	-
260°C /2 hr./6%S 1/8/99	5.33	-
Dry Fractionation of bleached Shea butter		
31°C Olein 8/27/98	5.23	9.71
31°C Stearin 8/27/98	11.08	14.30
Dry Fractionation of 50/50 Shea, sunflower blend		
28.3°C Olein 12/15/98	3.17	-
28.3°C Stearin 12/15/98	3.33	-
Bleached Shea butter	5.63	-
Sunflower oil	0.69	-
50/50 blend	3.16 ©	-
Wet Fractionation of 25%/75% Bl. Shea: hexane blend		
5°C Olein 7/13/98	6.86	9.65
5°C Stearin 7/13/98	2.28	2.24
Wet Fractionation with 95% by wt. ethanol		
Latex unsaponifiables	45.12 Ψ	
Shea butter from upper layer (olein)	8.30 Ψ	
Shea butter from lower layer (stearin)	4.68 Ψ	
Wet Fractionation with Abs. ethanol	Bl. Shea, 10°C	SM/Shea, 15°C
Latex unsaponifiables	44.31***	81.04***
Shea butter crystals when alcoholic solution cooled	0.53***	1.83***
Shea butter olein when alcoholic solution cooled	11.41***	9.86***

* - This method was selected and tested based on the high unsaponifiable content of the Shea butter relative to other non-marine oils. There were emulsion problems and hence phase separation problems with the non-marine oil method which may have elevated the measured unsaponifiable content.
** - data extracted from Table 1B-8
Ψ - data extracted from Table 1C-2
***- data extracted from Table 1C -4
© - Calculated

Phase 3C. Coco butter substitute (CBS)

The European Union has endorsed an agreement reached in June 1999 to allow chocolate products to contain 5% of certain vegetable fats other than cocoa butter as long as they are clearly labeled.

The agreement would allow six specific fats to be used instead of cocoa butter for up to 5% of the weight of the product, Illipe, Palm oil, Sal, Shea, Kokum, Gurgi and Mango kernel.

The physical properties of cocoa butter can be matched by blending the triglycerides, POP, SOS and POS all obtained from different sources. Shea butter is rich in the SOS triglyceride, Palm oil mid fraction is rich in the POP triglyceride while Illipe is rich in the POS triglyceride.

Three formulations were prepared using various combinations of these 3 oils and resulting formulation characteristic compared to two purchased commercial CBS. Results is displayed in Table 3C1, Table 3C2 and Figure 3C1.

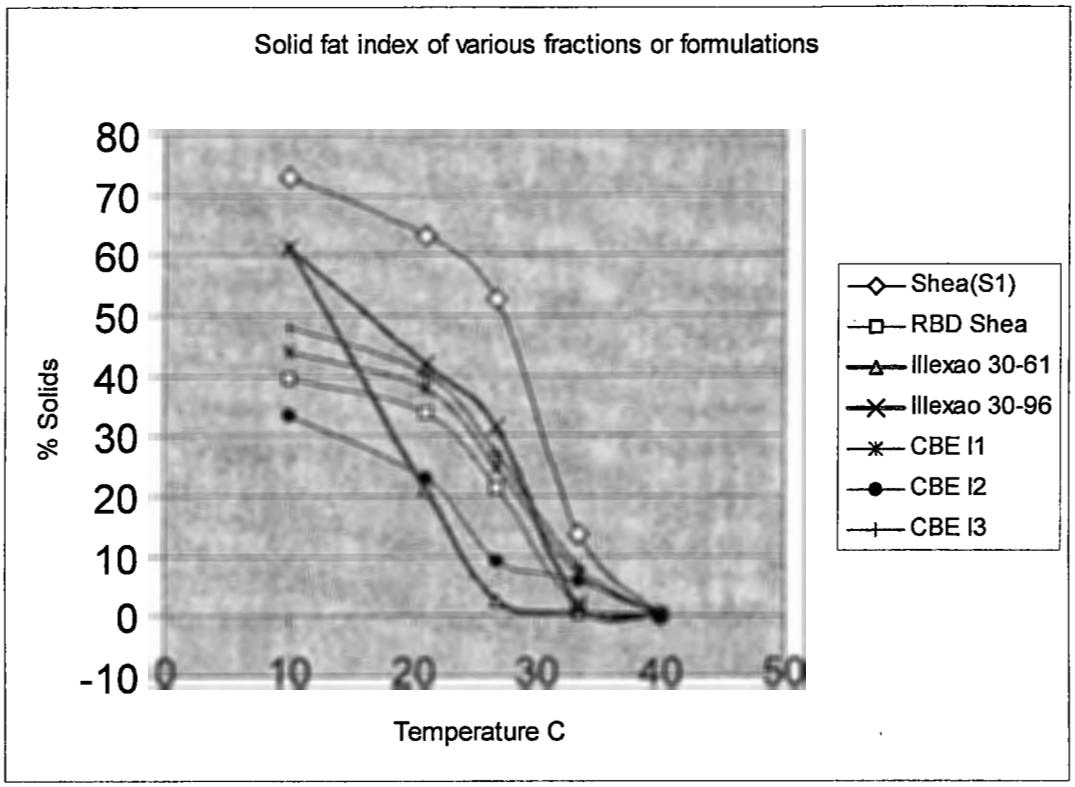
Table 3C1. Analytical data on formulation components and CBS formulations

	Sample ID	Comments	Unsap ,%	Sap. value	Iodine Value	Dropping point, °C
1	Illipe butter		-		33.17	35.6
2	PMF	Palm oil mid fraction	0.13	199.2	35.87	54.3
3	Shea (S1)	Shea ppte in abs EtOH, 10°C	0.53	-	39.14	34.9
4	SM/Shea #2		6.13	178.7	56.9	30.8
5	Illexao 30-61	Commercial CBE*	0.28	190.9	31-36	32-35
6	Illexao 30-96	Commercial CBS**	0.52	188.9	35-37	36-38
8	CBE $_{I1}$	49% Shea (S1), 31% PMF, 20% Illipe	-	-	36.48	40.9
9	CBE $_{I2}$	50% SM/Shea, 30% PMF, 20% Illipe	-	-	46.76	40.5
7	CBE $_{I3}$	60% Shea (S1), 40% PMF	-	-	-	42.5

Table 3C2. Solid fat Index of formulations

Temperature °C	Shea(S1) % solid	RBD Shea % solid	Illexao 30-61 % solid	Illexao 30-96 % solid	CBE $_{I1}$ % solid	CBE $_{I2}$ % solid	CBE $_{I3}$ % solid
10	72.95	39.50	61.15	61.17	43.88	33.60	47.84
21.1	62.99	33.93	20.86	41.76	37.71	22.96	40.29
26.7	52.59	21.26	2.36	31.38	24.82	9.29	26.92
33.3	13.53	1.44	0.59	1.65	6.71	5.69	8.35
40	0.00	0.00	0.00	0.00	0.00	0.00	0.00

Figure 3C1: Solid fat Index of various fractions and formulations



As can be seen from Table 3C-1 and Figure 3C-1 none of the three formulations had a Mettler dropping point close to the commercial CBE or CBS, i.e. 40-42°C compare to 32-38°C. This may be attributed to triglycerides with high melting point, 30-40°C, still present in the formulations. For instance the SFI curve of Shea stearin fraction, S1, clearly shows the high melting point components. Removing of the high melting point components from the Shea Stearin could consist of a double fractionation in ethanol. The first fractionation of RBD Shea could be done at a temperature, ~ 15-20°C . Any solids that precipitate out would be the triglycerides with high melting point.
The slurry should be filtered and the olein fraction re-fractionate at 10°C to yield a Shea stearin fraction devoid of the triglyceride with high melting point. Likewise the high Mettler dropping point of Palm oil mid fraction, (PMF) would suggest that this component of our formulation would have to be fractionated to remove any high melting point components before final blending in the formulation. The De Smet crystallizer and membrane filter in Burkina Faso will be indispensable in obtaining the various stearin fractions needed as the use of a membrane filter, which was not available at POS, will produce a stearin cake with a steeper SFI curve.

4A:Pilot plant scale-up for the refining & fractionation of Shea butter.

4A1:Introduction:

About one tonne of crude Shea butter was air freighted from Burkina Faso to POS. The crude oil was bleached and deodorized. Some of the refined oil was fractionated using hexane as the fractionating solvent. The olein and stearin fraction was fully desolventized and packaged.

4A2: METHODS:

Bleaching

The SM/Shea butter received from Burkina Faso was loaded into a reactor. It was liquefied and dried under vacuum. 0.2% by weight of citric acid was then added to the dried oil to chelate any metals that may be present. 6% by weight of adsorptive clay, Supreme 124FF, and 1% by weight of Darco activated carbon were then added to the oil for the removal of peroxides, phosphatides & color bodies. The slurry was heated to 110°C under vacuum for 30 minutes. The oil was then cooled to 65°C, vacuum broken with nitrogen, and filtered using filter aid. See Appendix 4A-1 for process directive.

Activated carbon was used in the scale-up trial due to the high initial colour of the starting butter. However it is rarely used in oil processing because of its high cost, high oil retention and handling difficulty.

Deodorizing

The bleached oil was sparged with steam at high temperature and low pressure to remove odoriferous components, flavour components, and additional free fatty acids. Color was also reduced by heat bleaching at elevated temperatures.
The oil was deodorized at 260°C in a continuous deodorizer using stripping steam at a rate of 1.0%/hr. The oil was cooled before it exits the deodorizer to 55°C.

The product was packaged under nitrogen into 20 liter pails after being fortified with 300ppm of dL-Alpha-Tocopheryl Acetate. See Appendix 4A-2 for process directive.

Crystallization

96 kilos of bleached and deodorized Shea butter was melted and loaded into a 300L reactor. 96 kilos of hexane was added and the slurry was heated to 50°C to destroy all crystals that might be present. The miscella was then cooled to 5°C and held at temperature for at least 10 hours. The waxes were then filtered using the Heinkel reversing basket centrifuge after addition of filter aid. Both the stearin and olein fraction were then desolventized and packaged in 16 kilo plastic pails. See appendix 4A-3 for process directive.

4A3: Methods - Analytical

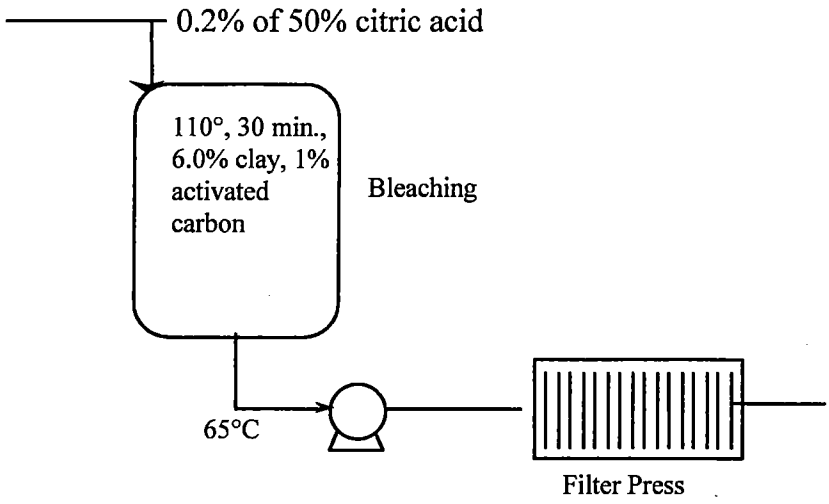
Peroxide Value, meq/kg	AOCS Cd 8-53
p-Anisidine Value	AOCS Cd 18-90
Free Fatty Acids, %	AOCS Ca 5a-40
Color	Auto Tintometer Lovibond Color, PFX 990 AOCS - Official Methods of the American Oil Chemists Society.
Unsaponifiables, %	AOCS Ca 6b-53
Residual hexane, ppm	AOCS Ca 3b-87

4A4:Discussion:

Bleaching

Prior to the pilot plant work a representative sample of the starting Shea butter was bleached and deodorized in the laboratory to determine the optimum amount of bleaching clay required to lower the colour in the starting Shea butter.
A lighter starting crude Shea butter colour would have significantly reduced bleaching clay requirements. Lower bleaching clay levels will reduce the loss of neutral oil in the bleaching process (normally 20-30% of clay weight).

Fig 4A1: Bleaching of Shea Butter.



Deodorization

Deodorization was successful in reducing the free fatty acid to an acceptable level of 0.09%, final colour of deodorized butter was 13.0Y1.7R. See Certificate of Analysis displayed in Table 4A1.
During deodorization different feed rates to the deodorizer and dwell time in the retention tank were tried in an attempt to optimize the deodorization process. Results are displayed in Table 4A2. The best conditions for the continuous deodorizer included a feed rate of 200 kg/hr with a dwell time of 16 minutes, (all valves closed in the retention tank), see Figure 4A2, to allow for maximum heat bleaching effect on the Shea butter.
If refining of Shea butter technology is to be transferred to Burkina Faso then atmospheric bleaching and batch deodorization will be the method of choice as the level of complexity of the equipment needed will be minimal. The colour of the final product will however be darker.

Fractionation of refined Shea in hexane

Fractionation of the refined Shea was done in hexane instead of ethanol as the fractions were to be tested on the cosmetic market in Canada. i.e. there was no need to obtain a very low unsaponifiable stearin fraction.
The trial yielded 42.1 kg of a stearin fraction with an unsaponifiable content of 6.15% and a Mettler drooping point of 33.7°C. The olein fraction, (20.1 kg)had an unsaponifiable content of 8.9% and a dropping point of 12.5 °C (Table 4A-4). The olein fraction with its creamy texture and high unsaponifiable value may be of interest for the cosmetic industry.

Certificate of Analysis

Bleached and deodorized Shea butter after antioxidants addition

POS Project number: 56-97-856

Production Date: January 2000

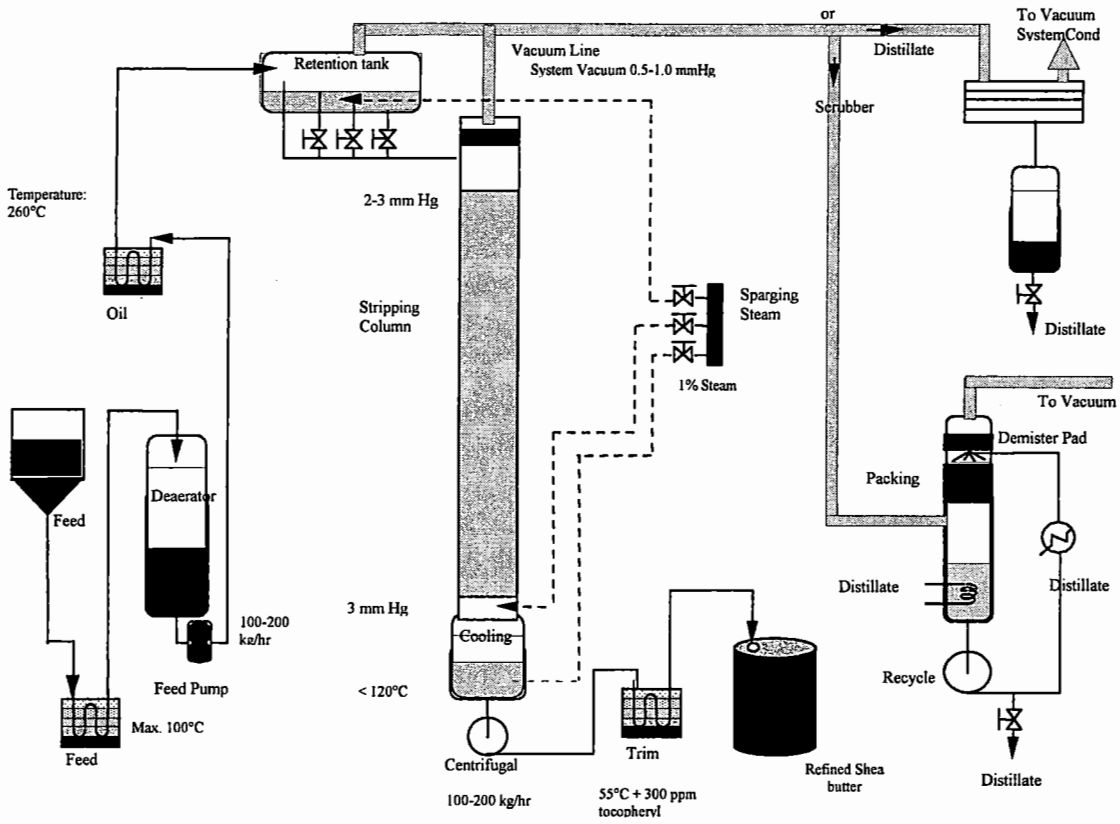
Table 4A1: OD3/out/Refined Shea oil after antioxidant addition

	Specification of commercial Refined Shea	Result	Method Reference
Peroxide Value, meq/kg	< 3.0	0.00	AOCS Cd 8-53
Free fatty acid %, (oleic)	< 1.0	0.09	AOCS Cd 3d-63
Moisture and volatiles, %	< 0.05	0.03	AOCS Ca 2d-25
p-Anisidine value	-	1.9	AOCS Cd 18-90
Colour 5 1/4" Lovibond(PFX990 AOCS)	-	13.0Y1.7R	POS Method SOP 3.1.9

Table4A2: Oil analysis during refining

Sample ID	PV, meq/kg	FFA, %	Colour AOCS	Notes
SM/Shea butter	2.15	6.52	20.0Y3.7R	Colour determined through 1" cell
OJ30A/S.cup/Bleached Shea butter	0.39	6.57	16.0Y1.7R	Colour determined through 5.25" cell
OD3/out/refined Shea	0.00	0.06	14.0Y2.2R	Feed rate to OD3 100 kg/hr, retention in retention tank, 32 minutes; 3 valves closed.
OD3/out/refined Shea	0.00	0.07	14.0Y2.1R	Feed rate to OD3 200 kg/hr, retention in retention tank, 16 minutes; 3 valves closed
OD3/out/refined Shea	0.00	0.07	24.0Y3.8R	Feed rate to OD3 200 kg/hr, minimum retention in retention tank, 6 minutes; all 3 valves open.

Figure 4A2:Continious deodorizer used for Shea butter deodorization.



4A5: Mass Balance:

Material balance for the pilot plant trial is displayed in Table 4A3. Material losses during processing were normal for the pilot plant scale given the initial dark colour of the Shea butter. Material loss during fractionation may seem high but is usually the case when processing small amounts of oil.

Table 4A3: Mass balance of pilot plant trial:

BLEACHING	
Input (kg)	Kg
SM/crude Shea butter	944.83
SM/citric acid(50%)	1.88
Bleaching Clay	56.68
Activated Carbon	9.40
SM/Filter aid	0.57
Total	**1,013.36**
Output (kg)	
F4/out/bleached Shea butter, ~910L X 0.9	819.0
F4/drain/Shea butter	11.5
F4/Frame Shea butter cake	151.0
Total	**981.5**
% oil loss	12%
DEODORIZATION	
Inputs, kg	
F4/out/bleached Shea butter, ~910L X 0.9	819.0
Tocopheryl, lot # 852078	0.216
Total	**819.2**
Outputs, kg	
OD3/out/transitin to Shea	36.02
OD3/out/refined Shea butter	711.60
OD3/out/shea butter drain	1.94
Total Outputs	**749.56**
Total Oil Loss, %	*8.5*
FRACTIONATION	
OD3/out/refined Shea oil, 6 pails	95.0
SM/Hexane	95.7
SM/filter aid	1.92
Total	**192.62**
Outputs, kg after 1st desolventization	
MP3/out/Shea Olein fraction	21.7
F1/out/Shea Stearin fraction	46.6
F1/Blow/Shea stearin	5.7
F1/frame/Shea Cake	4.0
F1/out/Shea line drain	2.4
Total Outputs	**80.4**
Total Oil Loss, %	*17.9*
Second desolventization	
MP3/out/Shea Olein fraction	21.7
F1/out/Shea Stearin fraction	46.6
Outputs, kg after 2nd desolventization	
MP3/out/Shea Olein fraction	20.1
MP3/out/Shea Stearin fraction	42.1

Table 4A4: Analysis of stearin and olein fraction of Shea butter.

Sample I.D.	Mettler D. point °C	Unsaponifiables %	Residual hexane ppm
OD3/out/Refined Shea oil	-	5.74	-
F1/out/Shea Stearin fraction	33.1	6.15	0.53
MP3/out/Shea Olein fraction	12.5	8.70	0.0

4A6: Conclusions

Quality of the starting Shea butter was marginal, with a relatively low peroxide value of 2.15 meq/kg, high free fatty acid content (6.52%) and dark oil color (20.0Y3.7R) as measured using a filtered Shea butter sample in a 1" cell.

The following materials were produced:

Table 4A5: Shea samples produced from scale up run.

Sample ID	Kilos
Refined Shea butter	711.6-95.0 = 616.6
F1/out/Shea Stearin fraction	42.1
MP3/out/Shea Olein fraction	20.1

Phase 4B. Commissioning of RBD Technologies Lab Deodorizer

4B1. Summary

The unit was received in July and the manufacturer spent 2 days assembling and demonstrating the equipment. Kassamba Bakari observed the working principles of the unit.

There were vacuum problems in the unit when lines on the vacuum/distillate side of the deodorizer plugged with Shea butter distillate. Heat tape and a hot air gun were used to resolve the problem during the initial trials with the Shea butter. RBD technologies redesigned the distillate side and supplied heat tracing and a redesigned distillate trap. These were received in late August 1999. The two major free fatty acids from Shea butter are Stearic acid (melting point 72°C), and Oleic acid (MP 16°C). The vacuum line was heat traced at a temperature of 70-75°C to prevent plugging with condensation of free fatty acids.

In the interim while waiting for the parts, trials were conducted using conventional soybean oil. These results are displayed in Table 4-1 (Operational Parameters) and Table 4-2 (RBD oil Quality)and compared very favourably with the same material when it was run in the pilot plant. The unit was tested with both nitrogen sparging and steam sparging and no significant difference in oil quality was observed, trials 8/21/98 and trials 8/24/98.

The first trials, 8/19/98 and 8/20/98, were learning runs. The technician had to get an understanding for temperature control during the initial trials, a small amount of cross contamination of previous materials through the column were encountered and staff were trying to get an estimate of how much oil at the start needed to be removed. (The initial feed over the column is off as a result of the stripping column not being wetted at the start of the test. This amounts to about 200-300 g of oil at the start.) The last trial with the soybean oil 8/25/98 was not as good because of a sticky water flow meter.

Our conclusion from these trials is that sparging with nitrogen is comparable to steam and to POS's pilot plant operation. The advantage of nitrogen over water in this unit is that the control of the stripping gas is considerably easier to regulate.

Table 4B-1. Commissioning of Deodorizer with Soybean oil - Operational Parameters

Date of trial	RB oil Mass (g)	% Recovery [a]	Time to Pressurize De-aerator to 14 psi (min.)	Oil Flow Meter Setting	Feed Rate (kg/hour)	Oil Loss due to Low Column Temp. (g)[b]	Deodorizer Heating Rate (°C /min.)	Variation Around Column Temp. Set point (°C)	Spirafilm Temp. Setting (°C)	Total Run Time (hours)[c]
Nitrogen Stripping gas 182-301 ml/min. Nitrogen flow varied to maintain system pressure at 2.0 mm Hg										
8/19/98	3219.5	96.67	30	60	2.0	70.0	3.1	259.0 - 265.3	194	5.0
8/20/98	3254.4	81.66	36	90	5.4	559.1	2.4	260.0 - 263.5	194	5.0
8/21/98	3232.5	92.82	31	60	2.0	169.3	3.0	258.0 - 265.3	194	5.3
1.0% Steam used for Stripping gas. System pressure 0.4 - 1.7 mm Hg										
8/24/98	3208.2	90.94	-	60	2.3	242.3	2.5	262.3 - 264.1	194	5.5
8/25/98	2958.2	88.91	28	90	5.2	223.7	2.6	258.0 - 262.0	196	4.6

[a] % Recovery : Deodorized oil (g) / RB oil Mass (g)

[b] Initiation of oil flow over the column decreases the column's temperature from 270°C to approximately 125°C , it takes 10 minutes for the temperature to rise back to 260°C . The oil collected prior to the column reaching 260°C was discarded as this oil was dark brown in color.

[c] The total run time includes the time it takes to check vacuum (~ 12 min.) and cool the oil to 100°C .

RB: Refined and bleached

Note:
1. The actual steam rate on 8/24/98 was 1.2% and on 8/25/98 was 1.1%.
2. In 25.4 hours of operation the deodorizer used 700 psi of nitrogen from a K size nitrogen bottle.
3. The maximum flow rate for water is 1.27 g/minute: therefore the maximum steam rate for a feed rate of 5.0 Kg/ hr is 1.5% and the maximum steam rate for a feed rate of 2.0 kg is 3.8%.

Table 4B-2. Commissioning of Deodorizer with Soybean oil - Analytical Results

Date of trial	Sample	Peroxide Value	% Free Fatty Acids	AOCS Color 5.25"		Taste (Randy Kruger)
				Red	Yellow	
8/17/98	RB Soybean oil	0.48	0.03	2.8	70.0	-
	RBD Soy-Pilot Plant	0.00	0.01	0.5	3.3	-
8/19/98	N_2 RBD Soy Flow : 60 Initial [a]*	0.40	0.01	4.6	22.0	-
	N_2 RBD Soy Flow : 60 Final [b]*	0.20	0.01	1.0	3.9	-
8/20/98	N_2 RBD Soy Flow : 90 Initial [a]	0.33	0.02	0.9	13.0	-
	N_2 RBD Soy Flow : 90 Final [b]	0.00	0.02	0.3	2.3	off taste
8/21/98	N_2 RBD Soy Flow : 60 Initial [a]	0.00	0.01	1.0	3.9	-
	N_2 RBD Soy Flow : 60 Final [b]	0.00	0.01	0.3	2.2	good
8/24/98	Steam RBD Soy Flow : 60 Initial	0.00	0.01	0.7	4.5	-
	Steam RBD Soy Flow : 60 Final [b]	0.00	0.01	0.4	2.4	good
8/25/98	Steam RBD Soy Flow : 90 Initial [a]**	0.20	0.02	1.5	14.0	-
	Steam RBD Soy Flow : 90 Final [b]**	0.00	0.01	0.8	3.4	good

[a] Initial - refers to a sample taken from the deodorizer 20 minutes after the column temperature stabilized between 260-265°C .
[b] Final - refers to a sample taken from the deodorizer after the oil in the collection vessel had been heated to 265°C for 20 minutes and then cooled to 100°C .
* These samples had a brown tinge.
** During this run the water flow meter's float became stuck and oil flow over the column was stopped until this problem was corrected . Once oil flow over the column ceased the Spirafilm heater over heated to ~ 300°C , this may account for the darker color of these samples.

Note: 1395.0 g of RB soybean oil was passed over the column prior to the series of trials to remove residues from the previous run, however the first trial samples (8/19/98) had a brown tinge to them. The second batch had good color but the taste was off. It therefore appears that more than 1,395.0 g of oil may be needed to pre-flush the deodorizer.

4B2. Commissioning of RBD Technologies Deodorizer after POS Modifications

The following modifications were made to the deodorizer to improve its ability to deodorize Shea butter.

1. Changed position of thermocouple on Spirafilm heater from middle of heater to top of heater so as to minimize overheating of oil in Spirafilm heater.

2. Installation of superior heating cable on the column. The new cable spirals down the column instead of an up and down arrangement. This will provide for more intimate contact between column and heating tape.

3. Installation of a -Z- pipe at the base of the column. This will provide for a means of diverting the initial and final oil flow over the column to a separate drain pot instead of the main deodorizer collection pot.

4. Installation of a drain pot between column and deodorizer for collection of start up and shut down oil.

5. Installation of a large distillate trap to enable batch deodorization of oil with high free fatty acid content.

6. Installation of an electrical element to liquefy distillate in the distillate trap at end of run.

7. Replace vacuum line with stainless steel tubing so that it may be heat traced.

8. Heat trace & insulate all vacuum lines to prevent FFA condensation in lines. This will lead to loss of vacuum if allowed to occur.

9. Construct a larger water reservoir for steam generation to resolve the problem of having to refill the reservoir during a run. The larger diameter reservoir also alleviates the problem of constant adjustment of steam flow rate during a run.

10. Installation of switch controlled power plugs for convenience of operation.

11. Place aluminum cladding on deaerator, column, and deodorizer for ease of cleaning and protection of insulation from oil, water damage.

Phase 5. Economic feasibility:

5A1. Overview

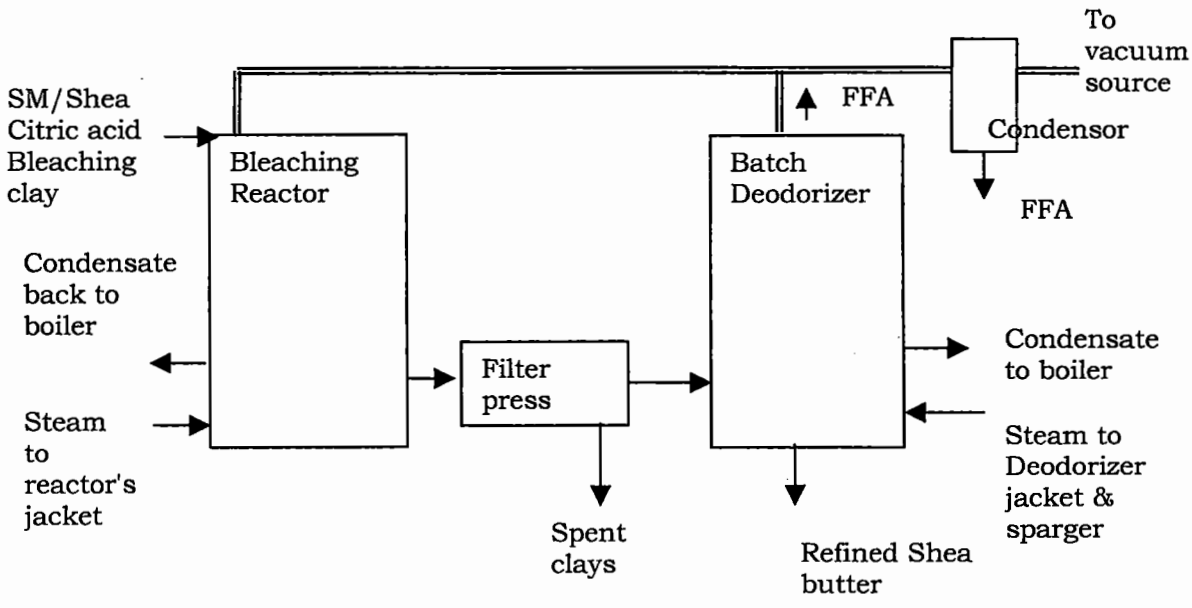
In view of the high variability of colour and free fatty acid level in crude Shea butter and low level of technological infrastructure in Burkina Faso it is proposed that any refining processing Shea plant, if built, will consist of a bleaching reactor, a filter press and a batch deodorizer.

Light coloured and odorless Shea butter produced from the refining process could compete as a cooking oil in Burkina Faso overcoming the main objection in using Shea butter as a cooking oil which is its strong odour. However, to achieve the lowest possible cost of production, all precautions should be taken to start processing with the highest quality of crude Shea butter. A good starting specification for crude Shea butter would be one with a free fatty acid content of 2% or less, a starting colour of 70.0Y3.0R or less and an oxidative state of 2.0 meq/kilo or less.

Starting with a butter having dark colour, high free fatty acid content and oxidative value will necessitate the use of high dosages of clay and possibly additional treatment with activated charcoal to bring down oxidative value and colour to acceptable levels. Lower colour and oxidative value will significantly reduce bleaching clay requirements, which in turn will reduce the loss of neutral oil in the bleaching process (normally 20-30% of clay weight). See Appendix 5A-4 & 5A-5 for an example of dependence of overall yield on starting Shea butter quality.

High free fatty acids in the crude Shea will also result in high loses during the deodorization process. The free fatty acids distillate could however be sold to the local soap industry.

5A2. Proposed processing flow for Burkina Faso



- An enclosed reactor was chosen over an open tank for bleaching as this will generate a lighter colour bleached oil and will result in a safer operation as operators will have little chance to be in contact with the hot oil produced during bleaching.

- Generation of steam at the boiler could use Shea outer coat, a by product which is generated when the Shea nuts are cracked prior to butter extraction.

- A batch deodorizer has been selected over a continuous deodorizer because of the low capacity of the plant envisioned, less than 100 metric tons per day and the possibility of high free fatty acid content in some of the starting Shea butter. A batch deodorizer will be better suited for Burkina Faso due to its simplicity in design and operation.

- Using a good starting butter will generate less spent clay effluents and thus minimize disposal problems. The spent clay could however be incorporated into spent Shea meal from the extraction process for animal feed.

- If there is a desire to produce Shea butter with low unsaponifiables for the CBS market then one could envision a flammable area where refined Shea butter will be fractionated using absolute ethanol. After cooling to 10-15°C, the precipitate would be filtered and desolventized to generate a Shea butter fraction suitable to be incorporated into a CBS formulation.

- Similarly the olein fraction could generate a liquid Shea oil at room temperature which may be suitable for the cosmetic market due to its high unsaponifiable content. The fractionation of Shea butter in ethanol is however a costly proposition as it would require a building and equipment rated for a flammable area and strict adhesion to safety regulations.

- Hexane could be used as the fractionating solvent if the stearin fraction is not required to have a low unsaponifiable value.

- Initial high cost of acquisition of solvent will have a quick payback time as the solvent will be reused for a long time as long as efforts are made to keep fugitive solvent loss to a minimum.

5A3. Price of finished good

The price per kilo of refined Shea butter ex POS is a function of the price of starting Shea, processing costs and overall refining yield. In general the more butter processed and the higher the quality of the starting Shea, the lower the overall price per kilo of finished product, see Appendix 4B3 and Table 4B1.

Results are based on the assumption of a refining yield of 78% for poor quality SM/Shea butter and 90% for high quality SM/Shea butter.

Table 5A1: Cost of finished goods FOB POS, (1999)

	Amount of SM/Shea butter processed	1 Tonne	2 Tonnes	4 Tonnes
	Poor quality SM/Shea butter			
1	Cost of SM/Shea ex B. Faso	$ 1,190		
2	Packaging	$ 262		
3	Transport to POS	$ 2,690		
4	Total SM/Shea, ex POS, (1+2+3)	$ 4,142	$ 8,284	$16,568
5	Processing cost, poor quality SM/Shea*	$10,650	$12,915	$19,915
6	Total, (4+5)	$14,792	$21,199	$36,483
7	Cost/kilo of finished good	**$18.96**	**$13.59**	**$11.69**
	Good quality SM/Shea butter			
8	Processing cost, good SM/Shea*	$10,520	$12,515	$19,415
9	Total, (4+8)	$14,662	$20,799	$35,983
10	Cost/kilo of finished good	**$16.29**	**$11.56**	**$10.00**

*: Value extracted from Appendix 4B3.

Table 5A2. Pricing on North American Market, (1999)

Description	Quantity ordered, kg	Price/kilo in Canadian funds*
Pressed & RBD Shea butter		
	25-75	$24.75
	100-375	$22.50
	400-720	$19.50
	720 up	$15.65

* Handling and transport cost will have to be added to these prices.

It is clear from the above two tables, 4B-1 and 4B-2, that processing and marketing of Shea butter in North American can be a lucrative proposition provided the batch size is increased to 4 tonnes or more and only high quality starting Shea butter is sent to POS. The same logic could be applied to a processing plant in Burkina Faso as long as actual local labour cost and equipment acquisition cost are used.

Phase 6. Acquisition of De Smet Fractionation Unit

This phase was executed.
Dr Rigobert Yamego was trained on the operation of a DeSmet fractionation unit in Belgium. The fractionation unit was then shipped to Burkina Faso.

Appendix 1A-1. Alkali Refining Trial Protocols

Trial #1

0. CRUDE OIL

1. ACID PRETREAT/REFINING

i. heat oil to 60°C
ii. add 0.2% of 85% phosphoric acid
iii. mix 30 minutes (2 min. with Polytron (high Shear mixer/homogenizer))
iv. add 16°Be NaOH to neutralize FFA's + 0.08% excess
v. mix 30 min.
vi. heat to 70°C
vii. centrifuge

NOTE: 1. After addition of acid, oil turned dark black in color.
2. After caustic addition a slight emulsion formed.

Starting Weight : 1381.4g Refined oil : 1295.1g Recovery : 93.75%
Soap stock : 99.68g

Starting Material FFA : 2.04%
After acid addition FFA : 2.74 %

Caustic Addition Rate : 4.24%

Soaps : 2192.24 ppm

2. WATER WASHING

i. heat to 75°C
ii. add 15% of 95°C water
iii. mix 15 min.
iv. centrifuge
v. measure soaps, if soaps higher than 50 ppm, repeat steps i. to iv.

NOTE: 1. An emulsion formed after water addition:

a. An attempt to break the emulsion by heating the oil to 95°C and holding at temperature for 30 minutes failed.

b. An attempt to break the emulsion by heating the oil to 95°C and adding 5, 10, and 15% salt and holding for 15 minutes failed.

c. An attempt to break the emulsion by heating the oil to 70°C and adding 5, 10, and 20% isopropyl alcohol and holding for 15 minutes failed.

2. Due to the emulsion problem this trial was aborted.

Trial #2

0. CRUDE OIL

1. ACID DEGUMMING

i. heat oil to 60°C
ii. add 0.2% of 50% **citric acid**
iii. mix 30 minutes (2 min. with Polytron(high Shear mixer/homogenizer))
iv. mix 30 min.
v. centrifuge

NOTE: 1. Almost no gums were removed.
2. Some water was still present in the oil after centrifugation.

Starting Weight : 1200.0g Refined oil : 1156.2g Recovery : 96.35%

Starting Material FFA : 2.04%
After Degumming FFA : 1.93%, Phosphorus : <0.2 ppm

2. ACID PRETREAT/REFINING

i. heat oil to 80°C
ii. add 0.2% of 85% phosphoric acid
iii. mix 30 minutes (2 min. with Polytron(high Shear mixer/homogenizer))
iv. add 16°Be NaOH to neutralize FFA's + 0.08% excess
v. mix 30 min.
vi. centrifuge

NOTE: 1. Some water was still present in the oil after centrifugation.
2. Some crystallization occurred as the oil cooled while centrifuging.

Starting Weight : 1103.3g Refined oil : 1024.6 Recovery : 92.87%
Soap stock : 93.20g

Starting Material, Degummed oil FFA : 1.93%
After acid addition FFA : 1.93 %

Caustic Addition Rate : 3.19%

Soaps : 472.5 ppm

2. WATER WASHING

i. heat to 75°C
ii. add 15% of 95°C water
iii. mix 15 min., very slowly
iv. centrifuge
v. measure soaps, if soaps higher than 50 ppm, repeat steps i. to iv.

NOTE: 1. Two minutes after water addition an emulsion formed:
a. An attempt to break the emulsion by heating the oil to 90°C and holding at temperature for 30 minutes failed.
2. The oil was then centrifuged and had cooled to 40°C during the 15 minutes.
3. Some water phase was removed by centrifugation but the emulsion was still present. Centrifugation was then done at 90°C. Emulsion was still present.
4. Added drying step to bleaching to remove water.

Starting Weight : 1024.5g Washed oil : 848.3g Recovery : 82.79%
1st Wash Water : 244.0 g
2nd Wash Water : 27.0 g

Soaps : 602.12 ppm

FFA : 0.28%

3. BLEACHING

i. load oil into Parr reactor
ii. dry the oil for 35 minutes at 80°C under vacuum
iii. cool the oil to 40-50°C
iv. add 0.1% of 50% citric acid and mix 15 minutes
v. add 2.0% Tonsil 'Supreme 120 FF' bleaching clay
vi. pull vacuum and heat to 110°C
iv. hold 30 min.
v. cool to 65°C
vi. filter, using filter aid

NOTE: 1. As a result of the high soap content, this trial was aborted after bleaching.

Starting Weight : 793.0 Washed oil : 744.7 Recovery : 93.91%

Soaps : 343.8 ppm
Peroxide Value : 0.54 meq/kg
FFA : 0.47%
AOCS Color 9.1Y 0.7R

Appendix 1B-1. Physical Refining Trial Protocols

0. CRUDE OIL

1. BLEACHING

i. load oil into Parr reactor
ii. heat the oil to 40-50°C
iii. add 0.0, 0.1, or 0.2% of 50% citric acid and mix 15 minutes
iv. heat the oil to 60°C
v. add Tonsil 'Supreme 120 FF' bleaching clay in dosage specified
vi. pull vacuum and heat to 110°C
iv. hold 30 min.
v. cool to 65°C
vi. filter, using filter aid

NOTE: Changes made to protocol are as specified in Tables.

2. DEODORIZE

i. load 800g into 2L glass deodorizer
ii. pull vacuum
iii. heat to 100°C
iv. begin steam addition at 6%/hr
v. continue heating until 250°C
vi. continue sparging for 1 hr at temperature
vii. cool to 70°C with sparging
viii. remove oil

NOTE: Changes made to protocol are as specified in Tables.

Appendix 1C-1. Solvent (ethanol) Refining Protocol

0. CRUDE OIL

1. REFINING

i. Heat Shea butter to 50-55°C
ii. Add 25% by volume of 80°C, 95% (by volume) ethanol
iii. Mix with gentle agitation for 15 minutes
iv. Transfer to 55°C convection oven and allow phases to separate

Observations:
1. After 30 minutes some phase separation occurred.
2. After 3 hours separation still incomplete.
3. After 24 hours further separation occurred, however emulsion was present.
4. Trial abandoned.

Appendix 1C-2. Refining with 95.6% (by wt.) of ethyl alcohol

A: Removal of latex unsaponifiables:

i. Transfer 125g of bleached Shea butter to a Parr reactor.

ii. Add 6 times the bleached Shea weight as 95.6% **(by wt)** ethanol into a Parr reactor.

iii. Connect the inner vessel of the second Parr reactor with the first Parr reactor via an in line filter fitted with a 0.5 micron Teflon filter paper(see Diagram 1C-2 at end of Appendix).

Note: A 1.0 micron filter paper will allow some of the latex to go through.

iv. Heat-tape the in-line filter, setting the tape temperature to 120-125°C.

Note: The first three trials were done at 95°C the last four were done at 120°C.

v. Ensure that there are no leaks in the system and that the first reactor is isolated from the in line filter (valve 2 close).

vi. Start heating the slurry in the first Parr reactor to 120-125°C (Final pressure in reactor will be around 40 psig).

vii. Maintain temperature of slurry at 120-125°C for 1/2 hour (gentle stirring).

viii. Pressurize the second vessel and the in line filter to same pressure as in the 1st reactor by opening valves 3 & 4. Close nitrogen valve once pressure is attained.

ix. Start filtration by opening the valve 2 connecting the first reactor to the in line filter while pressurizing the first reactor to 45-50 psig with nitrogen (valve 1) to initiate flow to the filter.

Note: As the second Parr reactor fills up the pressure in this vessel will increase while the pressure in first reactor will drop, once the pressure is the same as in the first reactor isolate the second reactor from the inline filter and discharge the second reactor into a 2 L beaker. Restart step ix until transfer is over.

x. After all the slurry has been transferred to the second vessel close the valves on both sides of the inline filter. Allow filter to cool.

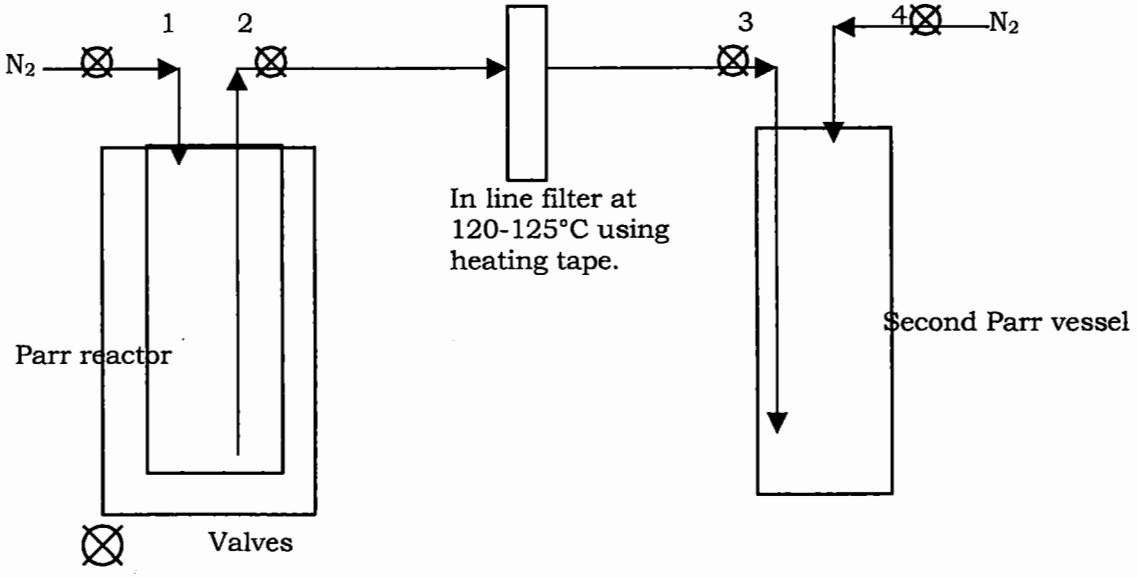
xi. Remove the filter paper from the inline filter.
Weight latex unsaponifiables on the filter paper after drying at 80°C for 1-2 hour.

Measure unsaponifiable, FFA after hexane extraction of the filter paper.
Note: Analysis for Latex unsaponifiables, upper & lower ethanol layer can be done by pooling 3 sample lot at a time.

B: <u>Alcohol refining (Removal of part of the FFA.)</u>

i. Transfer the filtrate from the second vessel once the temperature reaches 50°C into a separatory funnel. Allow to cool to 40°C.
ii. Hold at temperature for 1/2 hour.
iii. Decant the upper oil poor (and FFA rich layer) from the lower oil rich (and FFA poor layer).
Weigh both layers.
Measure % moisture of both layers (Karl Fisher).
iv. Desolventized the lower and upper layers separately using a rotary evaporator. Measure IV, saponification value, % FFA, and unsaponifiables of both layers.

DIAGRAM 1C-2 : Set up for alcohol latex removal:



Appendix 1C-3. Latex Removal by Absolute Ethanol Method

Protocol

SM/Shea butter or 2% clay atmospheric bleached Shea butter was mixed with absolute ethanol in a ratio of 1 part to 24 parts and heated to 80°C in a Roto-Vap. The resulting solution was mixed for 3.5 hours. The solution was then decanted from the precipitated latex that adhered to the wall of the Roto-Vap. The Shea butter-alcohol solution was cooled to 10°C (bleached) or 15°C (SM/Shea), and held at these temperatures for one hour. The crystals that precipitated out (S1) were separated from the ethanol-Shea solution by filtration, residual ethanol was removed from the crystals by drying in a vacuum oven at 65°C for 20 hours. The ethanol soluble oil fraction was then recovered by rotary evaporation, (O1).

Note: The 10°C ethanol soluble bleached Shea butter fraction, (O1) was desolventized in a Roto-Vap and then left at 22°C for 20 hours. Some crystallization occurred. The stearin(S2) and olein(O2) were separated by decanting the olein.

Appendix 3A-1. Dry Fractionation Trials

Using crude SM/Shea

6/12/98

-SM/Shea, (received 5/29/98) was filtered with Whatman #4 filter paper
-heat to 60°C then cool at 0.2°C /minute to **24.6°C and hold at temp. for one hour**
-very poor filtration, stearin solidified in the funnel

6/15/98

-SM/Shea, (received 5/29/98) was filtered with Whatman #4 filter paper
-heat to 60°C then cool at 0.2°C /minute to **28°C and hold at temp. for two hours**
-no crystals present at the start of the hold period
-at the end very small crystals were present but filtration was poor, the Whatman #5 filter paper plugged due to solidification
-since filtration was problematic it was decided to bleach the Shea butter prior to fractionation

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Using Bleached Shea

-the bleached Shea butter from the clay dosage trials done on the 6/16/98 was combined and used for the dry fractionation trials dated 6/17/98, 6/18/98, 8/27/98, 8/28/98 and 9/1/98.

6/17/98

-heat bleached Shea to 60°C then cool at 0.2°C /minute to **22.5°C and hold at temperature for 0.5 hours**
-stopped holding period after 0.5 hours because of excessive crystal formation
-filtration was possible but very little olein was recovered
-centrifugation was possible but very little olein was recovered
-since the holding time at 22.5°C was only 30 minutes, crystallization equilibrium probably had not occurred

6/18/98

-heat bleached Shea butter to 60°C then cool at 0.2°C /minute to **28°C and hold at temperature for 16.5 hours**
-after 16.5 hours the crystallization vessel temperature had fallen to 26.4°C , but the cooling water bath was still at 28°C
-complete crystallization had occurred, could not be filtered

8/27/98

-heat bleached Shea butter to 60°C then cool at 0.2°C /minute to **31°C and hold at temperature for 20.5 hours.**

Yield : 5.66% Stearin, Dropping Point of Stearin was 55.8°C , Dropping point of Olein fraction was 30.5°C .

9/2/98
-heat the 30.5°C olein to 60°C then cool at 0.2°C /minute to **29°C and hold at temperature for 19 hours.** Semisolid mass, could not filter.

9/3/98
-heat 31°C OL to 60°C then cool at 0.2°C /minute to **31°C and hold at temperature for 20 hours.** No crystals present.

8/28/98

-heat bleached Shea (produced on 8/28/98) to 60°C then cool at 0.2°C /minute to **29°C and hold at temperature for 16 hours.** Complete crystallization.

9/1/98

-heat bleached Shea (produced on 8/28/98) to 60°C then cool at 0.2°C /minute to **30°C and hold for 20 hours.** Lots of crystals, Tried to separate by centrifugation, not successful.

Shea/ Sunflower blend fractionation

12/10/98

300 g of 2% bleached Shea butter (produced on 9/22/98) was mixed with 300 g of RBD sunflower oil. The mixture was heated to 76°C and mixed for 15 minutes.

The blend was cooled to **25°C** using the DeSmet cooling curve assisted by agitation of 33 rpm **and held at temperature for 16 hours**.

	Yield, % By weight	Dropping Point (DP) °C
Stearin	45.32	31.5
Olein	50.58	13.5
Shea butter	-	31.8
Sunflower	-	-9.0

From the split between the stearin and olein fraction and dropping points it is concluded that almost all of the Shea had crystallized out.

12/14/98

The Shea/Sunflower blend was heated to 75°C and cooled to **18.5°C** with an agitation of 33 rpm **and held at temperature for 44 minutes.** After 30 minutes at 18.6°C complete crystallization occurred.

12/15/98

The Shea/Sunflower blend was heated to 75çc and cooled to **28.3°C** with an agitation of 33 rpm **and held at temperature for 20 hours.**
After 3 hours at 28.3°C crystal growth was observed (Oil turned cloudy).

	Yield, %	Dropping Point (DP) °C	IV	Unsaponifiables, %
Stearin	17.8	36.6	75.37	3.17
Olein	82.2	22.5	100.02	3.33
Shea butter	-	31.8	56.92	5.63
Sunflower	-	-9.0	135.25	0.69
50/50 Blend	-	-	-	3.16 *
3/23/99 Stearin	~ 20.0	33.3	50.02	-
3/23/99 Olein	~ 80.0	30.5	52.23	-

* calculated

Dry Fractionation of alcohol refined Shea (latex removed)

-a mixture of the 120°C alcohol refined bleached Shea was prepared as follows:
142.2g of bottom layer from trial 6 &7 (2/23/99) were combined with 112.9g bottom layer from trial 4& 5 (2/22/99).

3/12/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **31°C then add 0.2% seed crystals (5°C ST 7/16/98) and hold for ~ 48 hours.**
-due to evaporation from the water bath the temperature had fallen to 23°C . Complete crystallization had occurred.

3/15/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **31°C then add 0.2% seed crystals (5°C ST 7/16/98) and hold for 20 hours.**
-crystallization vessel temperature was 30.6°C . Complete crystallization had occurred.

3/16/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **31°C hold 2 hours then add 0.02% seed crystals (5°C ST 7/16/98) and hold for 1 hour.**
-before the addition of the seed crystals no crystallization had occurred, one hour after addition lots of crystals were present . No separation was achieved with the carver cell. Crystallization was probably occurring in the cell, and an excessive amount of crystals plugged the cell.
-note after 4 hours (2 hours after seed addition) at 31°C crystallization was complete.

3/17/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **35°C then add 0.02% seed crystals (5°C ST 7/16/98) and hold for 19.5 hour.**
-crystallization vessel temperature : 34.3°C . Complete crystallization had occurred.

3/19/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **36.6°C then add 0.02% seed crystals (5°C ST 7/16/98) and hold for 48 hour.**
-No crystallization had occurred.

3/22/99

-heat alcohol extracted Shea to 75°C then cool at 0.2°C /minute to **35.5°C then add 0.02% seed crystals (5°C ST 7/16/98) and hold for 20 hours**

3/23/99

-small amount of crystals present, crystallization vessel temp. = 35.8°C .
-some temp. variation in the vessel on the right side= 35.8 on the left =35.1 crystallization vessel temp. = 37.8°C .
-We increased the RPM to 84 (to equilibrate the temperature) cooled to 35°C held 23.5 hours.
-after 4.5 hrs. slightly more crystals, temp. now equilibrated but high agitation will result in small crystals, decrease RPM to 33.

3/24/99

-after 20 hours at 35.5°C and then 23.5 hours at 35°C a slurry of crystals was present
- Carver press-- separation of the larger crystals was possible but the smaller ones passed through the bottom and ST/OL was lost by up welling through the top of the press.
- Centrifuge at 4000 RPM for 10 minutes., ~20% ST was recovered

Appendix 4A-1: Process Directive of bleaching Shea butter in the Pilot Plant

Appendix 4A-2: Process Directive of deodorizing Shea butter in the Pilot Plant

Appendix 4A-3: Process Directive of fractionation of Shea butter in the Pilot Plant

Appendix 4B-1: Solving the darkening of the oil at start up

Trial #1: 2/22/99
Oil used: RBD canola oil (POS Startup oil)
Parameters :
Oil flow: 40% of rotometer scale
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Initial vacuum was 0.3 mm Hg.
The Spirafilm was set at 268°C with a variation between 265-271°C .
The column was set at 265°C with a variation between 263-266°C .
Spirafilm heater and column were heated to temperature **before** initiating oil flow to the column. After all the oil had passed over the column the deodorizer pot was heated to 265°C and held there for 20 minutes, the oil was then cooled to 100°C and the final product sample was taken. The first oil over the column was diverted to the drain pot. This oil was dark in colour. The final product was lighter in colour but was still darker than the starting oil. The deodorization process was darkening the oil.
Note: The cool down procedure of the Spirafilm heater and column was initiated once **all** the oil had passed over the column.

	Sample		
	Starting oil	Drain Pot oil	Final Product
Peroxide Value	0.8	0.4	0.2
% Free Fatty Acids	0.06	0.03	0.03

From the above data it appears that the increase in colour was not due to oxidation of the oil or free fatty acids. Oil colour may increase due to polymerization, oxidation, thermal degradation of phosphorus, or condensed fatty acids falling into the deodorized oil (vacuum loss). During this run the vacuum was good, therefore oxidation and fatty acid condensation should not have occurred. The oil had no phosphorus. Polymerization could have occurred just before cool down as the flow rate of oil over the Spirafilm and column would be tapering out at that time.

Trial 2: 2/24/99

Oil used: RB Palm oil
Parameters
Oil Flow rate: 40% of rotometer scale, i.e. Oil Rate of 2.27 Kg/hr.
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Hold deodorizer pot at 100°C until feed is finished then heat to 265°C and hold 20 minutes, then cool to 100°C and discharge.
Collect start up oil in drain pot until column temperature is at 260°C .
Note this oil had no phosphorus.
The Spirafilm was set at 269°C with a variation between 265-288°C .
The column was set at 265°C with a variation between 264-266°C .
Time to heat ~ 2900g of oil from 100-265°C was 55 minutes (3°C /minute).
The diverted drain pot start up oil was dark.

	Sample		
	RB Palm oil	Drain Pot oil	Final Product
Peroxide Value	0.00	-	0.00
% Free Fatty Acids	0.25	-	0.02
Colour 5.25" Lovibond [a]	70.0Y 18.1R	dark	34.0Y 3.0R

[a] The Lovibond scale was used because the oil was too dark for the AOCS scale.

It should be noted that as in the previous run (2/22/99) this oil had no phosphorus, the vacuum was good and the oil's temperature was kept below 300°C .
The dark start up oil was analyzed for polymerized and oxidized triglycerides, it was found to contain a 6.44% polar fraction (the usual value is 2%). The polar fraction contained 5.25% polymerized triglycerides and 17.17% oxidized triglycerides, therefore the dark colour was due to polymerization. Polymerization can occur at temperatures of 150°C and higher. During start up the column was heated to 265°C with no oil flow, the column was also cooled with no oil flow; this provided ample opportunity for the thin film of oil on the column to polymerize.

The start up and cool down was modified so that oil was flowing over the Spirafilm heater and the column during start up and cool down. The oil during start up and cool down was diverted to the drain pot as stripping of FFA would not be complete at lower temperature. When this procedure was followed we noted that the startup and cool down oil was no longer dark.

Trial #3: 3/3/99

Oil used: **Atmospheric bleached Shea butter**
Parameters
Flow: 40 % of rotometer scale
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Heating and cooling of the Spira heater and column was done with Shea butter flowing over these units.
Place ice and salt in the distillate trap to strip maximum amount of FFA before vapours reached the vacuum pump.
Hold deodorizer pot at 100°C until feed is finished then heat to 265°C and hold 2 hours. Cool to 100°C and discharge.
Collect start up oil in drain pot until column temperature is 260°C .
Note that this oil had no phosphorus.

Sample	% Free Fatty Acids	AOCS 5.25" Colour
SM/bleached Shea	2.03	9.9Y 0.6R
Drain Pot column at175°C	-	9.9Y 0.9R
Drain Pot column at 250°C	-	9.4Y 1.5R
Drain Pot column at 250°C	-	9.4Y 1.3R
Deodorizer at 265°C Time: 0	0.26	9.0Y 1.1R
Deodorizer at 265°C Time: 20 Min	0.19	9.0Y 1.2R
Deodorizer at 265°C Time: 60 Min	0.15	9.9Y 1.2R
Deodorizer at 265°C Time: 120 Min	0.05	8.5Y 1.1R

This trial demonstrated that the deodorizer can remove 97.5% of the Free Fatty Acids and that heat bleaching can reduce the yellow colour by 1.0 unit. It was also noticed that the red colour rose from 0.6 to 1.5; through heat bleaching this was reduced to 1.1.

The decrease in red seemed to occur when the temperature was around 265°C . It seems that some thermal degradation product in the atmospheric bleached Shea increased the red and this was partially removed during heat bleaching. The same behavior was observed when atmospheric bleached Shea was deodorized in a glass deodorizer.

Trial #4: 3/4/99

Oil used: **Atmospheric bleached Shea butter**
Parameters
Oil flow rate: 40% of rotometer scale.
Deodorization temperature: 265°C
Stripping gas: 3% steam
Note: Burkina Faso Clay was used instead on tonsil 120FF

Heat and cool the column with Shea butter.
When column temperature is less than 240°C collect oil in the drain pot. Place ice and salt in the distillate trap. Hold deodorizer at 100°C until feed is finished then heat to 265°C and hold 2 hours, then cool to 100°C and discharge. Collect oil in drain pot until column temperature is 260°C . Note that this oil had no phosphorus.

Losses during this run to heating and cooling : 870g, the deaerator was loaded with 2530.8g of Shea butter.

Sample	% Free Fatty Acids	AOCS 5.25" Colour
SM/BK. bleached Shea	1.70	9.9Y 0.8R
Deodorizer at 265°C Time: 0	0.02	10.0Y 1.4R
Deodorizer at 265°C Time: 1 hr	0.02	9.4Y 1.3R
Deodorizer at 265°C Time: 2 hr	-	9.7Y 1.4R

Once again deodorization increased the red. Heat bleaching did not occur for the red colour and only slightly for the yellow. The above Table shows that if oil passing over the column at temperatures below 240°C is discarded (start up and cool down oil) then a holding period in the deodorizer at 265°C is not necessary (this saves a minimum of 1.5 hours of operation time).

Trial #5: 3/5/99

Oil used: **Atmospheric bleached/Carbon Treated High FFA Shea butter.**
Initial colour of oil was very dark.
Note: This oil was first bleached with 2% clay , then it was treated with 0.5% carbon
Parameters
Oil flow rate: 60% of rotometer scale
Deodorization temperature: 265°C
Stripping gas: Nitrogen
Heat and cool the column with Shea butter.
When column temperature is less than 220°C **collect** oil in the drain pot. Place ice and salt in the distillate trap. Heat deodorizer to 265°C when feed is started, hold 2.5 hours at temperature after feed to the deodorizer has stopped, then cool to 100°C and discharge.

Sample	**% Free Fatty Acids**
SM/bleached High FFA Shea butter	13.14
Drain Pot - Column at 247°C	1.80
Deodorizer 240°C - Time : 0	0.28
Deodorizer 1 hour at 265°C	0.18
Deodorizer 1.5 hours at 265°C	0.17
Deodorizer 2.0 hours at 265°C	0.12
Deodorizer 2.5 hours at 265°C	0.08
Drain Pot -- Condensate from column during batch deodorization	0.12

During this run the distillate trap began to plug at the vapor inlet to the distillate trap. The plugging problem can be overcome by keeping the ice in the trap 12 cm below the top of the trap, also a heating tape could be placed around the top. In future runs the deodorization temperature should be kept at 100°C , if the deodorization temperature is above 180°C and vacuum is lost then fatty acids that have been distilled from oil in the deodorizer may fall back into the oil and affect the final colour. In the event of vacuum loss the flow from the column can be diverted to the drain pot, this should prevent unrefined oil from the column from falling into the deodorizer. The above procedure will increase operation time but should produce a better product.

It Is noted from the above Table that the column was able to remove 86.30% of the FFA present at a flow rate of 60% of rotometer scale, perhaps a slower flow rate would remove more.

Appendix 5A-1: Dependence of overall yield on starting Shea specifications

Processing Step	Assumptions	Theoretical Yield of low quality Shea Butter	Theoretical Yield of high quality Shea Butter
Bleaching	6% clay & 1% carbon dosage with low quality Shea Butter	88% *	-
	1.5% clay dosage with high quality Shea Butter	-	96% *
Physical Refining	6.52% of free fatty acid in low quality Shea Butter.	89.0% **	-
	2.0% max. free fatty acid in high quality Shea Butter.	-	93.5%**
Overall Yield Ψ		**78.3%**	**89.8%**

Ψ See Appendix 4B-2 for calculation.
* 30% by weight of entrained neutral oil in weight of bleaching clay used. See Appendix 4B-2.
** 1.0 % of neutral oil entrainment in deodorization. See Appendix 4B-2.

APPENDIX 5A-2 : Calculation of yields

Yield after bleaching:

With low quality Shea butter batch used in the pilot plant we use 6% clay dosage.

Yield after bleach = $\frac{\text{Wt of bleached Shea butter obtained in plant}}{\text{Wt of SM/Shea butter used at start of bleach}}$

$= \frac{819.00}{944.83} = 88.0\%$

If colour of SM/Shea butter was brought under control by proper processing in country of origin it is assumed that the amount of clay needed will be around 1.5% and that there will be no need for activated carbon.

Assuming 30% by weight of entrained neutral oil in weight of bleaching clay used and assuming a 20 kg filter press drain lost.

Yield, based on 1 tonne of SM/Shea after bleach will then be

$= \frac{(1000-(0.3 \times (1000 \times 0.015))-20}{1000} = 96\%$

Physical Refining Yield:

With a low quality Shea Butter the % of free fatty acid in the Bleached oil was 6.52%. Assume 1% neutral oil loss in deodorizer and 35 kg of transition oil at beginning of deodorization.

Yield, based on one tonne of SM/Shea after deodorization :

$$\frac{\text{Wt of deodorized oil}}{\text{Wt of bleached oil}} = \frac{\text{Wt of bleached oil - Wt of FFA- 1\% N. oil loss}}{\text{Wt of bleached oil}}$$

$$= \frac{1000 - 65.2 - 10.0-35.0}{1000} = 89.0\%$$

If the Shea seed were properly collected and stored a maximum free fatty acid content of 2.0% in the crude oil can be assumed.

Then the yield after deodorization will be: $\frac{1000 - 20.0 - 10-35.0}{1000} = 93.5\%$

OVERALL YIELD:

Overall yield for low quality Shea Butter:

88% x 89%= 78.3%

Theoretical Overall yield for Shea Butter with low colour and FFA, (2%):

96% x 93.5%= 89.8%

Thus if the starting material is top quality Shea butter approximately (898-783)= 115 kg of additional bleached and deodorized Shea butter per tonne processed will be achieved.

APPENDIX 5A-3. Calculation of costs at POS

A: Using low quality starting Shea butter

Estimated processing cost for refining SM/Shea butter with ~ 6% Free fatty acid content and dark colour, AOCS 20.0Y3.7R using 1" cell. An overall recovery of 78.3% has been assumed.

Amount of SM/Shea butter processed	1 Tonne	2 Tonnes	4 Tonnes
Labour at POS	$5,730.00	$6,600.00	$8,725.00
Material	$1,580.00*	$2,300.00	$3,300.00
Equipment	$3,300.00	$3,975.00	$7,850.00
Lab Supplies	$40.00	$40.00	$40.00
Total	$10,650.00	$12,915.00	$19,915.00
Processing cost per kilo of SM/Shea butter	$10.65	$6.46	$4.98
Processing cost per kilo of final RB Shea butter	$13.60	$8.25	$6.36

* See appendix 4B4 for estimation of material cost for processing 1 tonne of Shea butter.

High quality starting Shea butter.

Estimated processing cost for refining SM/Shea butter with ~ 2% Free fatty acid content and light colour, AOCS 70.0Y2.9R using 5 1/4" cell:
An overall recovery of 90% has been assumed.

Amount of SM/Shea butter processed	1 Tonne	2 Tonnes	4 Tonnes
Labour at POS	$5,730.00	$6,600.00	$8,725.00
Material	$1,450.00	$1,900.00	$2,800.00
Equipment	$3,300.00	$3,975.00	$7,850.00
Lab Supplies	$40.00	$40.00	$40.00
Total	$10,520.00	$12,515.00	$19,415.00
Processing cost per kilo of SM/Shea butter	$10.52	$6.25	$4.85
Processing cost per kilo of final RB Shea butter	$11.69	$6.95	$5.39

Appendix 5A4. Material usage for processing 1 tonne of low quality Shea butter

Material	Quantity used	Total Cost, $ Can.
Citric acid	1.88 kg	5.45
Bleaching Clay	56.68 kg	85.02
Activated Carbon	9.40 kg	57.37
Nitrogen		34.50
Filter aid	0.57 kg	0.74
Canola oil	170 kg	945.00
Pails & lids	46	257.70
Filter papers	16	38.40
Cartridge filters	2	56.00
Waste Disposal		50.00
Total		**$1,530.18**

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

Main Operators: _____ _____ _____ _____ _____ _____ _____ _____

Relief Operators: _____ _____ _____ _____ _____ _____ _____ _____

OBJECTIVE: To produce a bleached Shea butter product.

HAZARDOUS MATERIALS: Citric acid, bleaching clay, activated carbon, filter aid

GMP CONCERNS: Product is for human consumption. Hair nets and sanitary gloves to be worn when handling product.

RAW MATERIALS:

Quantity	Identification	Lot #	Container	Project #
~1000 kg	SM/Crude Shea Butter		boxes(50-55)	56-99-856
~2 kg	SM/50% Citric Acid	variable	PP	4100
~60 kg	SM/Supreme 124 FF Bleaching Clay	variable	bags	4100
~10 kg	SM/Darco Activated Carbon	variable	bags	4100
~0.6 kg	SM/Filter Aid	variable	bags	4100

EQUIPMENT

Main
CD, OP26, F4.

Auxiliary
PC26, OJ30A, scale for input materials.

Containers
Pails for drain and blow oil, open top drum for filter cake.

SETUP:

Step	QC Rep	Dev. (Y/N)	Procedure
1.0	Op ZK	NR	CD for softening butter.
2.0	Op ZK	N	OP26 for bleaching as per SOP. SIHI as vacuum source. Have man hole off for loading.
3.0	Op GK	N	F4 with 8 x 1" frames to filter the bleached oil.

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

Step	QC Rep	Dev. (Y/N)	Procedure
4.0	Op	N	PC26 to feed F4.
5.0	Op	N	F4/out recirc to OP26 and forward feed to OJ30A. Nitrogen sparge at F4 outlet.
6.0	Op	N	Cartridge filter(1 micron-lab provided) at F4 forward feed and blow outlet.
7.0	Op	N	OJ30A with cover, nitrogen sparge to collect the filtered oil.

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

Step	QC Rep	Date/ Time	Dev. (Y/N)	Procedure
		Jan. 18/00		
8.0	Op ZK	1830	N	Test all scales that will be used during process and record on the events sheet.
8.1	Op ZK	1830	N	Remove bag from box, cut bottom of bag and let butter fall into reactor. If it is difficult to remove the butter from bag try steps 9.0-12.0.
9.0	Op ZK	1830	Y	Load CD with 9 boxes per chamber ensuring there is space between boxes for air flow.
10.0	Op ZK	"	Y	Set temperature to 70°C and turn on Recirc fans and exhaust fan
11.0	Op ZK	"	Y	Let run for approximately 1 hour or until the butter appears to have softened considerably.
12.0	Op ZK	"	Y	Remove bag from box, cut bottom of bag and let butter fall into reactor.
13.0	Op ZK	"	Y	Repeat steps 9.0-12.0 until all butter has been loaded.
14.0	Op ZK	2100	N	Heat OP26 until butter is liquefied. Do not exceed 75°C while OP26 is not under vacuum.
				✓ **SAMPLE**
15.0	Op ZK	2130	N	Pull full vacuum and heat oil to 75°C ± 5°C.
16.0	Op ZK	2140	N	Hold for 30 min. or until there is no more bubbling.
17.0	Op ZK	2220	N	Cool the oil to 55°C. Break vacuum with nitrogen.
18.0	Op ZK	2305	N	Add 0.2% by wt of SM/50% citric acid.
19.0	Op ZK	2310	N	Hold for 15 minutes.

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

Step	QC Rep	Date/ Time	Dev. (Y/N)	Procedure
20.0	Op	1345	N	Add 6% bleaching clay and 1% activated carbon through top via funnel. Let dust settle for 5 min. then pull full vacuum.
21.0	Op	0115	N	Heat oil to 110°C ± 5°C and hold for 30 min.
22.0	Op	0210	N	Cool oil to 65°C ± 5°C.
23.0	Op	0217	N	Break vacuum with nitrogen and add 1% filter aid by wt. of clay.
24.0	Op	0230	N	Start feed to F4 and recirculate back to OP26 until clarity has been achieved and an additional 5 min. after that.
25.0	Op	0235	N	Forward feed to covered OJ30A. Have nitrogen on F4 outlet and a sparge on OJ30A.
26.0	Op	0310	N	At the end of the feed to F4 blow the press with Nitrogen for 30 minutes. If the oil flow out of F4 slows to a trickle sooner stop the blow at that time. Add the blow oil to the main batch if it is clear.
				SAMPLE
27.0	Op	0440	N	Break F4, weigh the cake and discard the cake after it has been wetted down. Label as: **F4/frame/Shea Butter cake.**
28.0	Op	0530	N	Collect all drain oil in a 20L pail and store in the warehouse. Label as: **F4/drain/Shea Butter**
~~28.0~~ 29.0	Op	0350	N	Store samples in secondary cooler.

POS
PROTEIN OIL STARCH

EVENTS

PROJECT 56-97856 **PAGE** 1 **OF** 2

OPERATION Bleach Shea Butter

TIME/DATE	INITIAL	EVENTS
1830 / Jan. 18 2000	ZK	Tested K6 w/ 10 Kgs - Scale reads 9.99 Kgs
		Weighing boxes of shea butter (Coats #'s not
		consistent, some have "pos" labels, others don't - will
		write down what is on boxes)
2115 / "	ZK	All butter is loaded, man hole back on, waiting
		for butter to liquify: using LP steam
2130 / "	ZK	Sample taken, pulling foil vac., continue heating
2140 / "	ZK	OP26 @ temp., hold for 30 mins
2220 / "	ZK	Cooling to 55°c
23:05 / "	RE	ADDING 50% citric Acid
2310 / "	ZK	15 min Acid hold
23:30 / "	RE	HOLD over ADDING CLAY + CARBON
2345 / -	RK	Pulling vacuum and heating to 110°C
0030 / Jan. 19 2000	RK	At temp. holding for 30 min
0115	RK	Hold over, start cooling to 65°C
0210	RK	At temp, breaking vacuum and adding filter aid
0220	RK	Start feed to F4 recircing until clarity
		is achieved
0225	RK	stop feed, hold in feed line
0227	RK	changed lines, start feed
0230	RK	clarity achieved, recircing for 5 min
0235	RK	Forward feed to 0530A
0237	RK	Forward feed oil will not get past the 1 micron
		filter. JS advised to take out the filter
0242	RK	start forward feed
0310	RK	End of feed blowing press
		- blow is clear, adding to tank

POS
PROTEIN OIL STARCH

EVENTS

PROJECT 56-97856 **PAGE** 2 **OF** 2

OPERATION Bleach Sinker Boiler

TIME/DATE	INITIAL	EVENTS
0440 / Jan. 19/00	RR	End of blow, breaking press

POS
PROTEIN OIL STARCH

INPUTS

PROJECT 56 97856 **PAGE** 1 **OF** 3

OPERATION Bleach Shea Butter

TIME/ DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
Jan. 18/00	0926	SM/ Crude Shea Butter	15/		17.28	ZH
			10/		16.50	
			28/		17.74	
			25/		17.88	
			3/		17.77	
			22/		17.82	
			29/		16.72	
			2/		16.55	
			9/		17.46	
			17/		17.80	
			18/		17.90	
			26/		17.63	
			22/		17.79	
			13/		17.80	
			14/		18.21	
			7/		17.97	
			10/		18.0	
			19/		17.79	
			20/		17.24	
			27/		17.75	
			23/		18.04	
			24/		17.64	
			5/		16.85	
			6/		16.72	
			8/		17.95	ZH
PAGE TOTAL					438.30	ZH

POS
PROTEIN OIL STARCH

INPUTS

PROJECT 56-97856 **PAGE** 2 **OF** 3

OPERATION Bleach Shea Butter

TIME/ DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
Cont'	0926	SM/Crude Shea Butter	9/		17.77	ZK
			3/		18.0	
			4/		17.74	
			46/		17.98	
			47/		17.23	
			48/		17.61	
			49/		17.90	
			53/		17.76	
			52/		16.62	
			55/		16.99	
			54/		16.39	
			51/		17.34	
			50/		17.77	
			95/		17.36	
			40/		17.56	
			44/		17.88	
			41/		16.67	
			43/		16.83	
			42/		16.99	
			38/		17.52	
			39/		17.89	
			34/		17.15	
			37/		18.14	
			6/		17.17	
			32/		17.80	
PAGE TOTAL					436.36	ZK

POS
PROTEIN OIL STARCH

INPUTS

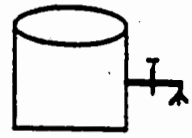
PROJECT 56-97856 **PAGE** 3 **OF** 3

OPERATION Bleach Shea Butter

TIME/ DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
cont'	OP26	SM/ Crude Shea Butter	34/		17.64	ZK
			29/		17.82	
			35/		16.87	
			30/		17.84	
		Sub-total Butter			944.83	ZK
Jan.18/00 2305	OP26	SM/ Citric Acid 50%	4/4	May26 '99	1.83	ZK
2330	"	BLEACHING CLAY			56.68	DK
"	"	ACTIVATED CARBON			9.40	DK
ORIG	"	SM/ Filter Aid			.57 ~~2.40~~ EL	DK
					1012.19	DK
PAGE TOTAL					~~74.17~~	ZK

POS
PROTEIN OIL STARCH

OUTPUTS



PROJECT 56-97856 **PAGE** 1 **OF** 1

OPERATION Bleach Shea Butter

TIME/ DATE STARTED	OUT OF	CONTAINER I.D.	CONT #/#	CONT TYPE	NET WT (kg)	INIT
0942 / Jan. 19/00	F4	F4/out/Bleached Shea Butter	1/1	0530A	~910 L	RR
0530	F4	F4/drain/Shea Butter	1/1	PP	11.5	RR
0940	F4	F4/Frame Shea Butter cake	1/1	MD cardb.	151.0	RR
					~910 L	RR
PAGE TOTAL					162.5 Kg	RR

Containers: Use Equipment I.D. or the following:

BS-Bulk sack	G-Garbage	MP-Metal Pail	PS-Paper Sack	CS-Cotton Sack
LB-Large Bucket	PD-Plastic Drum	SC-Sample Container	FD-Fiber Drum	MD-Metal Drum
PP-Plastic Pail	PT-Tote	PB-Porta Bin		

PROCESS DIRECTIVE

Project Number: 56-97-856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 18, 2000

PROCESS: Bleach Shea Butter

PROCESS DATA

Page 1 of 2

TIME (every 30 min or process change)		TARGET	2145	2215	2245	2315	2345
Operator	init		ZK	ZK	ZK	ZK	BK
OP26 temperature	°C		77.5	76.4	63.7	55.8	54.0
OP26 vacuum	"Hg		-23	-23.5	NC	Atm.	NA
F4 inlet pressure	Psi		NA	NA	NA	NA	NA
F4 rate	Pump setting		NA	NA	NA	NA	NA

TIME (every 30 min or process change)		TARGET	Jan. 19 2000 0015	0045	0115	0145	0215
Operator	init		BK	BK	BK	BK	BK
OP26 temperature	°C		92.9	109.6	109.5	93.6	67.3
OP26 vacuum	"Hg		-25	-27	-27	-27	NA
F4 inlet pressure	Psi		NA	NA	NA	NA	NA
F4 rate	Pump setting		NA	NA	NA	NA	NA

PROCESS DIRECTIVE

Project Number: 56-97-856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 18, 2000

PROCESS: Bleach Shea Butter

PROCESS DATA

Page 2 of 2

TIME (every 30 min or process change)		TARGET	Jan 19 2000 0945				
Operator	init		RV				
OP26 temperature	°C		62.3				
OP26 vacuum	"Hg		NA				
F4 inlet pressure	Psi		45				
F4 rate	Pump setting		Full				

TIME (every 30 min or process change)		TARGET					
Operator	init						
OP26 temperature	°C						
OP26 vacuum	"Hg						
F4 inlet pressure	Psi						
F4 rate	Pump setting						

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

PROCESS DATA

Page ___ of ___

TIME (every 30 min or process change)		TARGET					
Operator	init						
OP26 temperature	°C						
OP26 vacuum	"Hg						
F4 inlet pressure	Psi						
F4 rate	Pump setting						

TIME (every 30 min or process change)		TARGET					
Operator	init						
OP26 temperature	°C						
OP26 vacuum	"Hg						
F4 inlet pressure	Psi						
F4 rate	Pump setting						

PROCESS DIRECTIVE

Project Number: 56-97-856 January 18, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Bleach Shea Butter

PROCESS SAMPLES

SAMPLE I.D.	NUMBERxSIZE	DETAILS	TIMES/INIT
SM/Shea	1 x 500 ml	Take from OP26 after butter is liquefied.	21:35 / RZ
F4/out/Bleached Shea Oil	1 x 500 ml	Take from OJ30A after filtering is complete.	0425 /RZ

PROCESS DIRECTIVE

Project Number: 56-97-856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 18, 2000

PROCESS: Bleach Shea Butter

Deviations:

Step #	QC Rep	Date	Deviation(s)
9.0-13.0	ZK	Jan. 15 2000	These steps were not required

NOTE: THE NEXT PAGE IS EQUIPMENT AND MATERIALS CHARGES AND IS NOT ATTACHED TO THIS DIRECTIVE AFTER THE PROCESS ENDS.

PROCESS DIRECTIVE

Project Number: 56-97856 January 19, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Deodorize Bleached Shea Butter

Main Operators: ____ ____ JR ____ ____ ____ ____ ____

Relief Operators: ____ ____ ____ ____ ____ ____ ____ ____

OBJECTIVE: To produce deodorized Shea butter with a low fatty acid content.

HAZARDOUS MATERIALS: Have the scrubber discharge line going into a drum at all times when automatic system is in use.

GMP CONCERNS: Hair nets and gloves required during handling of product.

RAW MATERIALS:

Quantity	Identification	Lot #	Container	Project #
~ 400 Kg	SM/RBD Canola oil	unknown	IBC	stock
~ 1000kg	F4/out/Bleached Shea Butter		OJ30A	56-99856
~ 0.3 kg	SM/DL-Alpha Tocopheryl Acetate	852078	PP	

EQUIPMENT

Main

OD3 with Scrubber

Auxiliary

OJ30A, MJ9.5B, 3x 3 micron filters, C-60, K25A or B, K3, K.6, nitrogen sparges.

Containers

3 buckets and 9 clean used pails, ~ 50 new pails, ~25 tear-tab lids, ~ 25 pour-spout lids

SETUP:

Step	QC Rep	Dev. (Y/N)	Procedure
1.0	Op	N	Stocked desk and refuse container in immediate area.
2.0	Op	N	3 micron filter in permanent canister after deaerator.
3.0	Op	N	Hose and barrel sucker on deaerator inlet.
4.0	Op	N	IBC of canola oil on scale at deaerator inlet.
5.0	Op	N	OD3 outlet with sight glass, 0-150°C thermometer, 3 way valve, 3 micron filter, nitrogen sparge, sample port and line to MJ9.5B.

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

Step	QC Rep	Dev. (Y/N)	Procedure
6.0	Op	N	MJ95.B, covered with agitator and nitrogen sparge hooked up to low pressure steam.
7.0	Op	N	C-60 set up for packaging into pails out of MJ9.5B. 3 micron filter on pump outlet.
8.0	Op	N	Ensure OD4 and distillate condenser flanges closed, scrubber flange open, valve to OP26 closed.
9.0	Op	N	Hose and barrel sucker on scrubber inlet and hose out to waste drum on scrubber outlet.
10.0	Op	N	3 buckets and 9 clean used pails, POS supplied pails and lids

PROCESS DIRECTIVE

Project Number: 56-97856 January 19, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Deodorize Bleached Shea Butter

THIS IS A HARD OIL. DO NOT ALLOW TEMPERATURE TO FALL BELOW 50°C.

Step	QC Rep	Date/ Time	Dev. (Y/N)	Procedure
11.0	Op	Jan 19/00 03:05	N	Test scales before process starts. Record the weight of the test weight and the scale reading on the event sheet for all scales that will be used.
12.0	Op			Heat up OD3 as per SOP using SM/RBD Canola oil. Set parameters to the conditions listed in step 15.0 of this directive, except leave all retention valves open during heat up. **Take data during recirculation and transition.** During heat up check operation of cooling water at trim cooler so we can achieve a temp. of 60 C.
13.0	Op			Charge the scrubber with about 70kg of SM/RBD canola oil and heat up. Have the distillate temperature is set to 90°C.
14.0	Op	09:57	N	When parameters on OD3 and scrubber are achieved and stable, empty OD3 into plastic buckets. Label as: **OD3/out/start up oil**
15.0	Op	16:15	N	Start feed the **F4/out/Bleached Shea Butter** to OD3 and deodorize at the following conditions**: Project leader will be changing the feed rate and retention during the process-please note changes on event sheet.**

Deaerator temperature	65 ± 5°C
Feed rate	100 ± 2 Kg/hr initially
Trim heater outlet temp	260 ± 3 °C. Manual control
LH inlet temperature	As necessary to get trim heater outlet temp. Likely ~300°C
Top retention sparge	Minimal, enough to give a reading
Cooling section sparge	Minimal, enough to give a Reading.
Bottom column sparge	**1.0%** of oil flow (1 kg/hr at 100 Kg/hr oil flow)
Top vessel retention	All valves **closed** after transition (max retention)
Collection pot level	**~30L (1/3 full)** after transition
Collection pot temp	<120°C
Trim cooler outlet temp	55 ± 5°C
Heat tracing	ON
System vacuum	**< 7mm at bottom of column.**

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

Step	QC Rep	Date/ Time	Dev. (Y/N)	Procedure
16.0	Op	9:30	-V	As soon as oil is coming down the column start to collect transition in plastic pails for 30 - 35 minutes. **Keep deaerator and collection pot level low during transition.**
17.0	Op	9:30	V	After 30 - 35 minutes, close the retention valves, drain the collection pot and blow the trim cooler with nitrogen. Record weight of the transition. Save pails for client. Label as: **OD3/out/transition to Shea**
18.0	Op MK	1025	N	Allow a level to build in the dearator and collection pot.
19.0	Op	10:40	N	Collect the first 2 pails out of OD3 and recycle to the feed tank. Do not record these weights on the mass balance sheets.
20.0	Op	1615	N	Collect the deodorized Shea Butter in MJ9.5B. Have a nitrogen sparge on in line.
21.0	Op	1615	N	At the end of the feed from the dearator blow the line to the trim heater with nitrogen, open the retention valves over the time specified by the graph (if necessary), and discharge until the pumps cavitate, then blow out the trim cooler.
22.0	Op	17.35	N	Once all the deodorized Shea Butter has been collected add 0.03% tocopheryl to MJ9.5B and let it mix for 15 minutes.
23.0	Op	1930	N	Package oil into 20L plastic pails, sparging each pail with nitrogen before putting lids on. Package half of the oil with pour spout lids and the other half with tear-tab lids. Label as: **OD3/out/Refined Shea Butter.**
				SAMPLE
24.0	Op	19:20	✓	Record the weight of distillate collected if or when the scrubber discharges. Discard the distillate after weighing. Record on the output sheet as: **OD3/scrubber/Shea distillate**
25.0	Op	19:20	✓	Divert the paratherm and cool down OD3 using procedure for non vegetable oil. Collect, weigh, and discard cool down oil.
26.0	Op	19:20	✓	Shut down scrubber as per SOP.

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

PROCESS DATA

Page 1 of 3

TIME (every 30 minutes)		Run Target	07:30	8:00	9:10	9:40	10:10
Operator	Init		AA	DS	MK	DS	MK
Heat up/Transition/Feed			Heat up	H	T	F	F
Deaerator temperature	°C	65 ±5°C	78	84	72	62	70
Deaerator level	Liter		~40	~30	BELOW GAUGE	20	20
LH return press	Psi		123	121	116	115	113
Trim heater outlet temp	°C	260 ± 3	262	262	263	260	260
LH inlet temp	°C		272.6	272.5	272.8	272.4	272.1
System absolute pressure	KPa			N/A	N/A	N/A	N/A
Deodorizer feed rate	kg/hr	100 ± 2	100	100	100	100	100
Sparge steam flow- collection pot	kg/hr	1.0	1.0	1.0	1.0	1.0	1.0
Bottom of column temp	°C		226	235	230	190	220
Bot. of column abs pressure	mm Hg	< 7	2	1	1	1	1
Collection pot level		~1/3	1/3	1/3	BELOW GAUGE	MT	MT
Collection pot temp	°C		118	100	130	MT	MT
Finished oil flow	kg/hr		~100	100	~100	MT	MT
Trim cooler water rate	read.		1.4	.5	.1	OFF	OFF
Trim cooler outlet temp	°C	55 ± 5	NA	N/A	60	N/A	N/A
Distillate recirc. Temp	°C	90 ± 5	90.7	96.7	104.0	105.9	107.7
Distillate recirc. Rate		~1200	~1200	1200	1200	1200	1200
System vapour temp	°C		36	41	56	116	146
Barometric cond. Temp	°C	27 ± 3	27	27	27	27	27.5
Column top vapour temp	°C		84	92	111	112	117
Retention tank # of valves closed	#	3	0	0	0	3	3
First ret. Oil temp	°C		237	232	250	N/A	253
Column top abs pressure	mm Hg		3	3	3	2	3
spent cartridge filters	#		0	0	0	0	0
Collection tank volume	L		NA	N/A	N/A	N/A	N/A

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

PROCESS DATA

Page 2 of 3

TIME (every 30 minutes)		Run Target	10:40	11:10	11:40	12:15	1245
Operator	Init		MK	MK	MK	GS	MK
Heat up/Transition/Feed			F	F	F	F	F
Deaerator temperature	°C	65 ±5°C	71	71	71	75	75
Deaerator level	Liter		~20	30-40	30-40	~20	~20
LH return press	Psi		112	111	110	109	108
Trim heater outlet temp	°C	260 ± 3	260	260	260	260	260
LH inlet temp	°C		272.1	272.1	272.1	272.0	272.1
System absolute pressure	KPa		NA	NA	NA	N/A	NA
Deodorizer feed rate	kg/hr	100 ± 2	100	100	100	100	100
Sparge steam flow- collection pot	kg/hr	1.0	1.0	1.0	1.0	1.0	1.0
Bottom of column temp	°C		226	227	227	228	228
Bot. of column abs pressure	mm Hg	< 7	2	2	2	2	2
Collection pot level		~1/3	1/3	1/2	2/3	1/3	1/3
Collection pot temp	°C		101	104	105	116	116
Finished oil flow	kg/hr		100	120	130	100	110
Trim cooler water rate	read.		.3	.2	.2	.1	.4
Trim cooler outlet temp	°C	55 ± 5	56	57	56	69	58
Distillate recirc. Temp	°C	90 ± 5	107.8	108.8	102.9	90.0	90.7
Distillate recirc. Rate		~1200	1200	1200	1200	1200	1200
System vapour temp	°C		153	157	157	158	158
Barometric cond. Temp	°C	27 ± 3	27	27	27.5	26.5	26.5
Column top vapour temp	°C		126	130	132	133	134
Retention tank # of valves closed	#	3	3	3	3	3	3
First ret. Oil temp	°C		253	253	253	253	253
Column top abs pressure	mm Hg		3	3	3	3	3
spent cartridge filters	#		0	0	0	0	0
Collection tank volume	L		NA	NA	~75	~160	~220

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

PROCESS DATA

Page 3 of 3

TIME (every 30 minutes)		Run Target	1315	1345	1415	1515	1545
Operator	Init		M.K.	M.K	MK	S	S
Heat up/Transition/Feed			F	F	F	F	F
Deaerator temperature	°C	65 ±5°C	73	73	74	80	76
Deaerator level	Liter		20	30	40	50	30
LH return press	Psi		107	106	106	106	106
Trim heater outlet temp	°C	260 ± 3	260	255	258	258	258
LH inlet temp	°C		272.1	281.7	284.4	288	288
System absolute pressure	KPa		NA	NA	NA	NA	NA
Deodorizer feed rate	kg/hr	100 ± 2	100	200	200	200	200
Sparge steam flow- collection pot	kg/hr	1.0	1.0	2.0	2.0	2.0	2.0
Bottom of column temp	°C		229	232	233	234	234
Bot. of column abs pressure	mm Hg	< 7	2	2	2	2	2
Collection pot level		~1/3	1/3	1/3	1/2	< 1/3	< 1/3
Collection pot temp	°C		117	128	124	136	129
Finished oil flow	kg/hr		110	200	210	200	200
Trim cooler water rate	read.		.6	.6	.5	.5	.5
Trim cooler outlet temp	°C	55 ± 5	65	62	64	50	58
Distillate recirc. Temp	°C	90 ± 5	90.8	91.2	91.0	91.0	90.5
Distillate recirc. Rate		~1200	1200	1200	1200	1200	1200
System vapour temp	°C		159	166	170	168	168
Barometric cond. Temp	°C	27 ± 3	26	26	26.5	26.5	27.0
Column top vapour temp	°C		135	145	150	148	150
Retention tank # of valves closed	#	3	3	3	3	0	0
First ret. Oil temp	°C		253	250	252	252	250
Column top abs pressure	mm Hg		3	3	3	3	3
spent cartridge filters	#		0	0	0	0	0
Collection tank volume	L		~270	~350	~450	~650	~740

PROCESS DIRECTIVE

Project Number: 56-97856
Project Leader: Herve Douce
Process Technician: Greg Gervais

January 19, 2000

PROCESS: Deodorize Bleached Shea Butter

PROCESS DATA

Page ____ of ____

TIME (**every 30 minutes**)		**Run Target**					
Operator	Init						
Heat up/Transition/Feed							
Deaerator temperature	°C	**65 ±5°C**					
Deaerator level	Liter						
LH return press	Psi						
Trim heater outlet temp	°C	**260 ± 3**					
LH inlet temp	°C						
System absolute pressure	KPa						
Deodorizer feed rate	kg/hr	**100 ± 2**					
Sparge steam flow- collection pot	kg/hr	**1.0**					
Bottom of column temp	°C						
Bot. of column abs pressure	mm Hg	**< 7**					
Collection pot level		**~1/3**					
Collection pot temp	°C						
Finished oil flow	kg/hr						
Trim cooler water rate	read.						
Trim cooler outlet temp	°C	**55 ± 5**					
Distillate recirc. Temp	°C	**90 ± 5**					
Distillate recirc. Rate		**~1200**					
System vapour temp	°C						
Barometric cond. Temp	°C	**27 ± 3**					
Column top vapour temp	°C						
Retention tank # of valves closed	#	**3**					
First ret. Oil temp	°C						
Column top abs pressure	mm Hg						
spent cartridge filters	#						
Collection tank volume	L						

PROCESS DIRECTIVE

Project Number: 56-97856   January 19, 2000
Project Leader: Herve Douce
Process Technician: Greg Gervais

PROCESS: Deodorize Bleached Shea Butter

PROCESS SAMPLES

SAMPLE I.D.	NUMBERxSIZE	DETAILS	TIMES/INIT
OD3/out/Refined Shea Butter	1 x 250ml	Take half way through packaging.	Jan.19/00 1830 / ZK
OD3OA/S.cup / Bleached Shea Butter	1 x ~~250~~ 100 ml		10:32 Jan19/00 CL
OD3/out/refined Shea Butter 3 valve closed, 100 Kg/hr	1 x 250mL		11:25/MK
OD3/OUT/REFINED SHEA BUTTER 3 VALVES CLOSED, 200 Kg/HR.	1 x 250 ML		14:00/MK
OD3/OUT/REFINED SHEA BUTTER 3 VALVES OPEN, 200 Kg/HR.	1 x 250 ML		1530 / S.

POS
PROTEIN OIL STARCH

EVENTS

PROJECT 56 97856 **PAGE** 1 **OF** 2

OPERATION Deodorize Bleached Shea Butter

TIME/DATE	INITIAL	EVENTS
03:00/Jan.19/00	M	Start parathem (set point 300°C)
03:05/ "	M	Test scale K.6 - 1kg reads 1.0kg
		- 10kg " 10.0kg
		- 20kg " 20.0kg
04:15/ "	M	Pull vac on ODS.
05:00/ "	M	Start up oil into deavator
05:20/ "	M	Taking long time to heat up, LH was open to SD1, shut value.
05:23/ "	M	Start flow to spray nozzle, filter canister for leaking gaskie.
05:33/ "	M	O ring was torn, replaced filter canister, charging scrubber with ~70 kg canola.
05:05/ "	M	Start discharge pumps
8:25	M.	start collecting Start up oil
8:57	M	Blowing Trim cooler, Start Feed to Deavator
9:07	M	start collecting transition
9:30	M	Closing Retention Values & blowing out trim cooler
9:33	M	Blowing Trim Cooler
10:25	M.K.	COLLECTING FIRST 2 PAILS OUT OF ODS AND RECYCLING BACK TO OT30A
1045	M.K.	COLLECTING IN MJ9.5B NOW
13:30 25th/02	M	Adjust feed rate to 2cwt/hr, steam sparge to ~~5~~ still be 1% of feed rate. All valves closed.
1332	M.K.	INCREASED LH SET POINT TO 300°C - THE TRIM HEATER OUTLET TEMP. DROPPED TO 247°C ~~AFT~~ RIGHT AFTER INCREASING FEED RATE TO 200 kg/HR.

POS
PROTEIN OIL STARCH

EVENTS

PROJECT 5697856 **PAGE** 2 **OF** 2

OPERATION DEODORIZE BLEACHED SHEA BUTTER

TIME/DATE	INITIAL	EVENTS
14:30/Jun 8/00	[illegible]	Stopping Feed to Trim heater, & Deaerator so
		that can open the retention values & Start the
		3rd Trial with values open. Opening retention
		Values.
14:42	[illegible]	As per PL request will also Blow the Trim
		cooler before starting Feed again
1510	MK	BLOWING TRIM COOLER, RE-START FEED AT 200 kg/hr
		ALL RETENTION VALVES OPEN
1533	[illegible]	end feed to deaerator.
1559	[illegible]	end feed to top - blowing
1615	[illegible]	blowing trim cool.
1620	[illegible]	start feeding cooldown/flush.
1650/ "	ZH	M39.5B has 800 L, 0.45 kg/500 mL = 0.90 kg/L
17:30	JP	Finish blowing trim cooler & shutdown oil
17:35	AG	added tocopheryl
1750/ "	ZH	Start packaging into P.P's @ 16.0 Kgs each
19:20	JP	Pumping out final granules shutdown oil.
		Shutting down ODT
19:30	AG	Finished packaging

POS
PROTEIN OIL STARCH

INPUTS

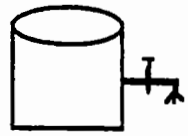
PROJECT 56 97856 **PAGE** 1 **OF** 1

OPERATION Deodorize Bleached Shea Butter

TIME/ DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
08:30/15-Jan-00	OD3	SM/RBD Canola Oil	1/1		100.0	AG
08:33/ "	Scrubber	" "	1/1		70.0	AG
8:57	OD3	F4/out/bleached Shea Butter	1/1	Jan19/00	~910 L	CG
1620	OD3	OD3 OUT START UP OIL	1-2/2		75.87	
1638	"	SM/ RBD CANOLA OIL	1/1		100.8	JR
17.35	MJ9.5B	Tocopheryl .216kg Lot# 852078	1/1	Nov.17 '98	.216kg	AG
18:00	Scrubber	SM/ CANOLA	1/1	-	150.6	JR
PAGE TOTAL						

POS
PROTEIN OIL STARCH

OUTPUTS



PROJECT 56 97856 **PAGE** 1 **OF** 1

OPERATION Deodorize Bleached Shea Butter.

TIME/ DATE STARTED	OUT OF	CONTAINER I.D.	CONT #/#	CONT TYPE	NET WT (kg)	INIT
06:00/Jan 19/00	OD3	OD3/Flush/Canola oil	1/1	PP	120	BL
08:35	OD3	OD3/out/start-up oil	~~1/2~~	LB	75.87	/
9:07	OD3	OD3/out/transition to Shea	1/2	PP	17.93	CS
9:22	"	" "	2/2	"	18.09	CS
	~~OD3~~	~~OD3/out/REFINED SHEA~~ Jan. 19/2000 EE				
1630	OD3	OD3/OUT/COOLDOWN FLUSH	1/	DISCARD MD	195.0	JA
1614	OD3/	OD3/Scrubber/Distillate Shea	1/1	MP	117.7	JA
					164.4	
19:00	Scrubber	Scrubber/out/Shutdown oil.	1/1	MD	~~195.0~~ JA	JA
1750	OD3	OD3/out/Refined Shea Butter	1-44/45	P.P	704.0	~~ZK~~
	"	" "	45/45	"	7.60	~~ZK~~
2000	OD3	OD3/out/Shea Butter/Drain	1/1	"	1.94	~~ZK~~
PAGE TOTAL						

Containers: Use Equipment I.D. or the following:

BS-Bulk sack	G-Garbage	MP-Metal Pail	PS-Paper Sack	CS-Cotton Sack
LB-Large Bucket	PD-Plastic Drum	SC-Sample Container	FD-Fiber Drum	MD-Metal Drum
PP-Plastic Pail	PT-Tote	PB-Porta Bin		

Appendix 4A-2: Process Directive of deodorizing Shea butter in the Pilot Plant

PROCESS DIRECTIVE

Project No.: 300-99856 Sept 13, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

Main Operators: _____ _____ _____ _____ _____ _____ _____ _____

Relief Operators: _____ _____ _____ _____ _____ _____ _____ _____

OBJECTIVE: To desolventize shea stearine fraction to less than 100 ppm of hexane.

HAZARDOUS MATERIALS: Hexane

GMP CONCERNS: Hairnets and gloves required while handling the product.

RAW MATERIALS

Quantity	Identification	Lot #	Container	Project #
~ 47 kg	F1/out/Shea stearine fraction		3 Pails	
~ 22 KG	MP3/out/Shea oleine fraction		2 Pails	

EQUIPMENT

Main
MP3

Auxiliary
Flammable single stage eductor.

Containers
5 new pails and tear tab lids

PROCESS DIRECTIVE

Project No.: 300-99856 Sept 13, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

SETUP : PROCESS TO TAKE PLACE IN FLAM 2.

Step	INITIAL	Dev. (Y/N)	Procedure
1.0	Op		Stocked desk and refuse container.
2.0	Op		MP3 with agitator extension set up for desolventization in Flam 2. Have sparger installed (the longest one). Agitator on variable speed (via extension cord to Flam 1 pot).
3.0	Op		Flammable single stage eductor as vacuum source with knock out pot in line.
4.0	Op		All submerged fittings to be soft gaskets.
5.0	Op		Nitrogen connected to MP3 sparger via nitrogen regulator.
6.0	Op		Vac gauge connected to MP3. (Valve between gauge and reactor)..
7.0	Op		Temperature gauge, tested, (0-150°C) on vacuum outlet to monitor vapour temperature.
8.0	Op		Temperature gauge, tested, (0-100°C) in MP3 **bottom side** port.
9.0	Op		Temperature gauge, tested, (0-100°C) in MP3 **bottom** port. Ensure this probe only extends ~ 3 inches into the bottom of the tank.
10.0	Op		Hose and barrel sucker connected to MP3 bottom port.

PROCESS DIRECTIVE

Project No.: 300-99856 b
Process Leader: Herve Douce
Process Technician: Greg Gervais g
PROCESS: Desolventize Shea Butter

Sept 13, 2000

PROCESSING

Step	INITIAL	Date/ Time	Dev. (Y/N)	Procedure
11.0	Op	7000	N	Put the 3 pails of **F1/out/shea stearine fraction** under a tarp and melt it using low-pressure steam.
12.0	Op	0920	N	Pull vacuum on MP3.
13.0	Op	0935	N	Suck in the **F1/out/shea stearine fraction**.
14.0	Op	0945	N	Start a fairly turbulent nitrogen sparge, start agitator at medium, and heat the liquid to 90°C ± 2°C.
15.0	Op	0100	N	Hold under vacuum until 0800 hours at 90°C ± 2°C. Ensure the nitrogen sparge and agitator are on during the hold.
				SAMPLE (desdired hexane level is less than 10ppm)
16.0	Op	1020	N	When analysis is good, cool to 50°C and break vacuum.
17.0	Op	1028	N	Using slight nitrogen pressure, feed to new pails with tear tab lids via 3 micron filter. Fill pails to **16.0 kg± 0.1 kg**. Sparge each pail with nitrogen for 2 minutes before sealing. **Label as: MP3/out/Shea stearine fraction.**
				SAMPLE
18.0	Op	1053	N	Rinse MP3.
19.0	Op	1115	N	Pull vacuum on MP3
20.0	Op	1115	N	Suck in the **MP3/out/shea oleine fraction**.
21.0	Op	1120	N	Start a fairly turbulent nitrogen sparge, start agitator at medium, and heat the liquid to 90°C ± 2°C.
22.0	Op	1125	N	Hold under vacuum for 2 hrs at 90°C ± 2°C. Ensure the nitrogen sparge and agitator are on during the hold.
				SAMPLE (desdired hexane level is less than 10ppm)

PROCESS DIRECTIVE

Project No.: 300-99856 Sept 13, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

Step	INITIAL	Date/ Time	Dev. (Y/N)	Procedure
23.0	Op CH	1600	N	When analysis is good, cool to 50°C and break vacuum.
24.0	Op CH	1625	N	Using slight nitrogen pressure, feed to new pails with tear tab lids via 3 micron filter. Fill pails to **16.0 kg± 0.1 kg**. Sparge each pail with nitrogen for 2 minutes before sealing. **Label as: MP3/out/Shea oleine fraction.** **SAMPLE**

PILOT PLANT CORP.

EVENTS

PROJECT 300-99856 **PAGE** 1 **OF** 1

OPERATION Desolventize Shea Butter

TIME/DATE	INITIAL	EVENTS
2000/Sept13/00	[illegible]	putting 3 pails of FI/out/steam fraction under saran plastic with low press. steam. Pails are located in Flambé filter room.
Sept14/00/0030	[illegible]	filling vac on MP3
0035	[illegible]	had to fix some N_2 leaks on lines to sparger, will start to suck oil in
0045	[illegible]	Oil all in, accidentally spilled ~1l of oil on floor; starting N_2 and agitator - start to heat up
0100	[illegible]	At temp; will maintain till morning.
0450	[illegible]	increased variable speed to max.
0510	[illegible]	switched MP3 to Normal speed as variable speed is needed elsewhere
0835	[illegible]	The next 2 pails have been put under plastic to melt.
1020	[illegible]	Analysis is good cooling reactor
1028	[illegible]	Start packaging
1052	[illegible]	Done Packaging / Rinsing Reactor
1115	[illegible]	Loading Reactor
1120	[illegible]	Start N_2, agitation + heating
1125	[illegible]	Oil at temp. Start 2hr. hold.
1330	[illegible]	Sample taken
1600	CA	LAB ANALYSIS OK - START COOLING TO 50°
1685	CA	AT TEMP - MP3 PRESSURIZED - START PACKAGING
1630	CA	END PACKAGING

POS
PROTEIN OIL STARCH

INPUTS

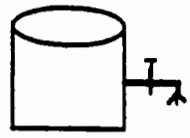
PROJECT 300-99856 **PAGE** 1 **OF** 1

OPERATION Desolventize Shea Butter.

TIME/ DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
0035	MP3	Fl/out Shea Sterine Fraction	1/3	Sept 6/00	16.0	DJ
—	"	" "	2/3	"	16.0	D.T
—	"	" "	3/3	"	14.6	DJ
111.5	MP3	MP3/out/Shea Olein Fraction	1/2	Sept 1/00	16.1	AFC
		" "	2/2	Sept 1/00	5.6	MPL
			~~1/2~~		~~16.1~~	~~CH~~ (ES)
			~~2/2~~		~~4.1~~	~~CH~~ (ES) ~~ES~~
PAGE TOTAL					68.3	CH

POS
PROTEIN OIL STARCH

OUTPUTS



PROJECT 300-99856 **PAGE** 1 **OF** 1

OPERATION DESOLVENTIZE SHEA BUTTER

TIME/ DATE STARTED	OUT OF	CONTAINER I.D.	CONT #/#	CONT TYPE	NET WT (kg)	INIT
1028	MB	MP3/OUT/ shea stearine fraction	1/3	PP	16.1	
1038	MP3	" "	2/3	"	16.1	
1045	"	" "	3/3	"	9.9	
1625	"	MP3/OUT/SHEA OLEINE FRACTION	1/2	"	16.0	
	"	" / / " "	2/2	"	4.1	
PAGE TOTAL					62.2	

Containers: Use Equipment I.D. or the following:

BS-Bulk sack	G-Garbage	MP-Metal Pail	PS-Paper Sack	CS-Cotton Sack
LB-Large Bucket	PD-Plastic Drum	SC-Sample Container	FD-Fiber Drum	MD-Metal Drum
PP-Plastic Pail	PT-Tote	PB-Porta Bin		

PROCESS DIRECTIVE

Project No.: 300-99856
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

Sept 13, 2000

PROCESS DATA

Page 1 of 2

TIME (every 30 minutes)		Target 0030	0100	0230	0300	0330	0400
Operator	Init	D.L.	DT	DT	DT	DT	DT
MP3 temp	°C	88	90	89	90	90	89
MP3 vapor temp	°C	54	55	56	57	58	57
MP3 vacuum	"Hg	-21	-20	-20	-20	-20	-20
Nitrogen sparge on		ON	ON	ON	ON	ON	ON

PROCESS DATA

TIME (every 30 minutes)		Target 0430	0500	0530	0600	0630	0700
Operator	Init	DL.	DT	DT	DT	DL.	DT
MP3 temp	°C	89	91	90	89	88	89
MP3 vapor temp	°C	57	59	59	59	57	58
MP3 vacuum	"Hg	-20	-19	-19	-19	-18	-18
Nitrogen sparge on		ON	ON	ON	ON	ON	ON

PROCESS DATA

TIME (every 30 minutes)		Target 0730	0830	0900	1000	1125	1155
Operator	Init	DT	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
MP3 temp	°C	92	95	90	90	90	93
MP3 vapor temp	°C	60	55	50	58	57	58
MP3 vacuum	"Hg	-18	-20	-18	-19	-18	-18
Nitrogen sparge on		ON	ON	ON	ON	ON	ON

PROCESS DIRECTIVE

Project No.: 300-99856 K
Process Leader: Herve Douce
Process Technician: Greg Gervais S
PROCESS: Desolventize Shea Butter

Sept 13, 2000

PROCESS DATA

Page 2 of 2

TIME **(every 30 minutes)**		**Target**	1235	1305	1335	1355	1415
Operator	Init						
MP3 temp	°C		90	85	90	88	80
MP3 vapor temp	°C		55	46	52	50	45
MP3 vacuum	"Hg		-18	-17	-18	-18	-18
Nitrogen sparge on			ON	ON	ON	ON	ON

PROCESS DATA

TIME **(every 30 minutes)**		**Target**	1515	1545			
Operator	Init						
MP3 temp	°C		73	85			
MP3 vapor temp	°C		50	48			
MP3 vacuum	"Hg		-18	-18			
Nitrogen sparge on			ON	ON			

PROCESS DATA

TIME **(every 30 minutes)**		**Target**					
Operator	Init						
MP3 temp	°C						
MP3 vapor temp	°C						
MP3 vacuum	"Hg						
Nitrogen sparge on							

PROCESS DIRECTIVE

Project No.: 300-99856
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

Sept 13, 2000

PROCESS SAMPLES

SAMPLE I.D.	NUMBER x SIZE	DETAILS	TIMES/INIT
MP3/out/desolventized shea stearine	1 X 100ml	Take to lab for analysis.	0813
MP3/out/Shea stearine fraction	2 x 250 ml	Take while pailing.	
MP3/out/desolventized shea oleine	1 X 100ml	Take to lab for analysis.	1330
MP3/out/Shea oleine fraction	2 x 250 ml	Take while pailing.	

PROCESS DIRECTIVE

Project No.: 300-99856
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Desolventize Shea Butter

Sept 13, 2000

Page 1 1 1

Deviations:

Step #	INITIAL	Date	Deviation(s)

Appendix 4A-3: Process Directive of fractionation of Shea butter in the Pilot Plant

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 1 of 9

Main Operators: _____ _____ _____ _____ _____ _____ _____ _____

Relief Operators: _____ _____ _____ _____ _____ _____ _____ _____

OBJECTIVE: To winterize and cold filter the Shea Butter.

HAZARDOUS MATERIALS: Hexane, Filter Aid.

GMP CONCERNS: Hairnets and gloves required while handling the product.

RAW MATERIALS

Quantity	Identification	Lot #	Container	Project #
~ 96 Kg	RBD Shea Butter		6 PP	
~ 4 Kg	SM/Filter aid		Paper bag	stock
~ 96 Kg	SM/Hexane		MD's	

EQUIPMENT

Main
MR7, BC2, MJ4, MP3, F1

Auxiliary
PC15, D30, K3, PVE, scale suitable for weighing filter aid

Containers
about 6 new pails and tear tab lids, open top drums for BC2 solids.

SETUP

Step	INITIAL	Dev. (Y/N)	Procedure
1.0	Op	N	Stocked desk and refuse container.
2.0	Op	N	MP3 with agitator extension set up for desolventization in Flam 1. Agitator on variable speed (via extension cord to Flam 1 pot).
3.0	Op	N	MP3 agitator seal charged with SM/canola oil.
4.0	Op	Y	130-psi pressure release valve installed on MP3.
5.0	Op	N	Portable 3-stage eductors for vacuum source with knock out pot in line.

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter

Step	INITIAL	Dev. (Y/N)	Procedure
6.0	Op	N	All submerged fittings to be soft gaskets.
7.0	Op	N	Nitrogen connected to MP3 steam sparger via nitrogen regulator.
8.0	Op	N	Vac gauge connected to MP3. (Valve between gauge and reactor.) **Do not install any portable mercury manometers.**
9.0	Op	N	Temperature gauge, tested, (0-150°C) on vacuum outlet to monitor vapour temperature.
10.0	Op	N	Temperature gauge, tested, (0-100°C) on MP3 **side** port.
11.0	Op	N	Plastic gloves and respirators in the area.
12.0	Op	N	Hose and barrel sucker connected to MP3 bottom port.
13.0	Op	N	Scale for weighing hexane drums.
14.0	Op	N/A	D30 for transferring MP3 contents to MR7 (pump will also be used for feeding BC2).
15.0	Op	N	BC2 with teflon filter bag. **(Install during day shift.)**
16.0	Op	N	MR7 with valve on bottom. Agitator plugged in and glycol connected. Valve on top for recirc. Temperature gauge (-10 to +10°C range) in MR7.
17.0	Op	N	D30 to feed BC2 from MR7, with recirc line back to feed tank via the dedicated automatic 3-way valve. Flexible line and sightglass from 3-way to BC2 inlet.
18.0	Op	N	BC2 h.out to red open top drum.
19.0	Op	N	BC2 l.outs to tared MJ4 via large stainless pipe. Reduce to put 3" sight glass in at lower level. MJ4 to have lid and vented.
20.0	Op	N	F1 with 10 frames, set up in the area. 3-micron filter on forward feed and blow outlet.
21.0	Op	N	Ground all equipment. Vent all tanks via wall fan and BC2 with slight suction only.

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 3 of 9

PROCESSING

Step	INITIAL	Date/ Time	Dev. (Y/N)	Procedure	
22.0	Op	Aug 30/00 1800	N	Put the 6 pails of RBD Shea Butter under a tarp and melt the butter using low-pressure steam.	
23.0	Op	2250	N	Pull vacuum on MP3 and then close vacuum line.	
24.0	Op	2300	N	Load MP3 with 96 kg ± 0.5 kg of SM/hexane.	
25.0	Op	2305	N	Heat the Hexane to 50°C ± 2°C.	
26.0	Op	2315	N	Suck in the 6 pails of RBD Shea butter and agitate well.	
27.0	Op	23:50	N	Break vacuum on MP3 and transfer the slurry to MR7. Have slow agitation on MR7.	
28.0	Op	23:50	N	Start cooling the slurry in MR7.	
29.0	Op	23:50	N	MR7 Agitator speed	10 ± 1 rpm
30.0	Op	23:50 Aug 31/00	N	Glycol set point	3 °C
31.0	Op			Final Product Temp.	5°C.
32.0	Op	23:00	N	Hold time at temp.	10 hrs at 5°C.
33.0	Op	Aug 31/00 00:40	N	Start timing the hold time when MR7 temperature reaches 5 °C.. Rinse MP3 with hot water and drain well.	
34.0	Op	15:42	N	When the winterizing hold is over do a five minute spin down of the material in MR7. Record the results.	
35.0	Op	15:50	N	Add **1.0% by slurry wt. of SM/filter aid** to MR7. Mix in well but do not increase agitator speed.	

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 4 of 9

Step	INITIAL	Date/ Time	Dev. (Y/N)	Procedure
36.0	Op	16:00	N	Using D30 as feed pump, separate using BC2 with the recipe indicated below to start. Adjust conditions for maximum throughput, **while maintaining l.out clarity and fairly dry solids.**

D30 air press	**20-25 psi**
Max. fill time	**300 seconds**
Max. fill weight	**40 Kg**
Min. fill weight	**35 Kg**
Fill speed	**750 rpm**
Dewater speed	**750 rpm**
Dewater time	**45 seconds**
Spin speed	**1900 rpm**
Spin time	**360 seconds**
Discharge speed	**500 rpm**

Notes: -Watch near the end of the filling part of the cycle to ensure that the bag is not being overloaded.

Check clarity of l.out regularly, especially after increases in spin speed.

37.0	Op	Aug31 1630	N	Collect the h.outs in new red open top drum. Make notes on the event sheet to describe the heavy out solids. Weigh the product. **Label as: BC2/h.out/shea butter solids. (DO NOT DISCARD)**
38.0	Op	1630	N	Collect the l.outs in MJ4. Weigh the product. **Label as: BC2/l.out/shea butter miscella.**
39.0	Op	1745	N	With steam and slow agitation on to MP3, add the BC2/h.out/shea butter to MP3 via sightglass port.
40.0	Op	1800	N	Seal MP3, increase agitation, and pull a full vacuum using all stages on portable 3-stage eductor.
41.0	Op	1800	N	Start nitrogen to sparger and heat the solids to 60°C ± 2°C.

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 5 of 9

Step	INITIAL	Date/ Time	Dev. (Y/N)	Procedure
42.0	Op	Aug 31/00 14:50	Y	Hold for 3 hours at 60°C ± 2°C.
				SAMPLE (desdired hexane level is less than 10ppm)
43.0	Op	Sept 01 12:40	N	When analysis is good, cool to 50°C and break vacuum.
44.0	Op	12:54	N	Feed to F1via PC15 and recirc to MP3 until clarity is achieved.
45.0	Op	13:00	N	Forward feed to new pails with tear tab lids. Fill pails to 16.0 kg± 0.1 kg. Sparge each pail with nitrogen for 2 minutes before sealing. **Label as: F1/out/Shea stearine fraction.**
46.0	Op	13:14	N	Blow F1 for 30 minutes or until there is only a trickle coming out. Add to the main batch if it is clear. If blow is not clear keep it separate and label as: **F1/blow/Shea stearine fraction.**
				SAMPLE
47.0	Op	15:36	N	Break F1, weigh the cake and discard. Record as **F1/frame/shea cake.**
48.0	Op	14:55	N	Pull vacuum on rinsed MP3 and suck in the BC2/l.out/Shea butter.miscella.
49.0	Op	15:07	N	Pull full vacuum, start medium agitation and nitrogen sparge, and heat miscella to ~~60~~°C ± 2°C. 90°C (APC) & 1 Sept 2000
50.0	Op	15:55	N	Hold for 3 hours at ~~60~~°C ± 2°C. 90°C (APC) & 1 Sept 2000
				SAMPLE (desdired hexane level is less than 10ppm) After hold (APC) & 1 Sept 2000
51.0	Op	18:25	N	When analysis is good, cool to 50°C and break vacuum. (APC) &
52.0	Op	18:50		NOTE: USE A 3 MICRON FILTER IN LINE FOR PACKAGING. 30 AUG 2000 Package into new plastic pails with tear tab lids using slight nitrogen pressure in MP3. Fill pails to 16.0 kg± 0.1 kg. Sparge each pail with nitrogen for 2 minutes before sealing. **Label as: MP3/out/Shea oleine fraction.**
				SAMPLE

PILOT PLANT CORP.

EVENTS

PROJECT 300-99856 **PAGE** 1 **OF** 3

OPERATION Crystallize, Filter and Desolventize Shea Butter

TIME/DATE	INITIAL	EVENTS
1805 Aug 30		Melting 6 pails of Shea butter
2300		Starting in hexane
2305		Heating to 50°C
2315		Loading Butter
~~23~~ 23:50		Begin transfer to MR7 using pressure - Cooling on to MR7
00:30 Aug 31/00		Cannot find MR7 Dipstick so cannot record Volume on Delta,
2:42		Decreasing set point to -5°C on bars to try & get to hold temp faster
03:25		Not mixing well or cooling fast enough. Adjusting Agitator RPM to 11.
4:20		will put agitator to Max. only ~ 11.5 - 12 RPM. to try & get to mix better
4:55		The outside edge of MR7 (along jacket walls) is 5°C while in the middle is ~9.0 - Turning Bars chiller back to 3°C for set pt.
07:00 ~~5:00~~		As per PT start hold as 05:00
5840		Temp 3.4 on outside and 5.2 in middle, will move Bars set point to 4° as per P.T's advise.
15:50		Add Filter aid to MR7 - Spin down was 3.6/15 = 24%
16:00		Start feed to BC2.
1630		FIRST Load came out fairly damp but the next load came out wet
		Decreased load size down to 20 kg

PILOT PLANT CORP.

EVENTS

PROJECT 300-99856 **PAGE** 2 **OF** 3

OPERATION Crystallize, Filter & Desolventize Shea Butter

TIME/DATE	INITIAL	EVENTS
1745 Aug31/00	[illegible]	Sucked the LOATS into MP3
1800	[illegible]	Start heating to 60°C
1850	AA	MP3 @ 60°C hold under full vac until morning. Drain K.O. pot.
21:00	AA	Shoot ~ 40 kg from original staking material, open BC2 to recover any product. Breaking vac on MP3 in order to add through sight glass.
21:10	AA	Recovered 5.2 kg from BC2, adding to MP3. Scraped the interior
00:00	CR.	Holding with slight N° sparge and full vac pulled (~25") at 60-62°C. Agitator at 35% on variable speed. Will hold till AM. at these conditions.
Sept01/00 10:50	AG	PL advises to heat up to 90°C and hold for an hour.
10:54	[illegible]	@ temp will hold for 1 hour.
11:37	[illegible]	PL requested sample be taken now.
12:40	[illegible]	cooling to 50°C
12:54	[illegible]	@ temp start feed to F1
13:00	[illegible]	oil looks good starting to boil.
13:14	[illegible]	and feed start to blow F1 with N2
14:55	[illegible]	sucking BC2/L.OUT/Shea Butter miscella and heating to 90°C
15:35	[illegible]	@ temp start 3hrs hold.

PILOT PLANT CORP.

EVENTS

PROJECT 300-99856 **PAGE** 3 **OF** 3

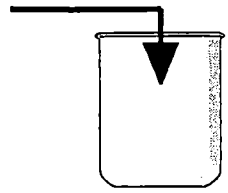
OPERATION Crystallize, Filter and Desolventize Stea Butter

TIME/DATE	INITIAL	EVENTS
1835 Sept 1	JDL	3 hr. hold over Breaking vacuum and cooling to 50°C
1850	JDL	Packaging
1920	JDL	Done packaging

Revised: April, 2000

PILOT PLANT CORP.

INPUTS



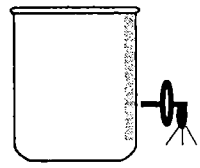
PROJECT 300-99856 **PAGE** 1 **OF** 1

OPERATION Crystallize, Filter and desolventize Shea Butter

TIME/DATE STARTED	IN TO	CONTAINER I.D. (FROM THE CONTAINER LABEL OF MATERIAL USED)	CONT #/#	DATE D/M/Y	NET WT (kg)	INIT
2300	MP3	SM / Hexane			95.7	
2315	MP3	OD3/out/Refined Shea 0.1 @ 96 kg		Jun 19/00	95.0	
15:50	MP3	SM / Filter Aid	NA	NA	1.92	
17:48 / 20:10	MP3	BC2/2.out/Shea Butter solids	1/2	Aug 3/00	<108.0>	
20:10 / 11	"	" "	2/2	"	<5.2>	
Dept of 14:55	"	BC2/2.out / SHEA BUTTER MISCELLA	1/1	Aug 3/00	45	
PAGE TOTAL						

PILOT PLANT CORP.

OUTPUTS



PROJECT 300 99 856. **PAGE** 1 **OF** 1

OPERATION Crystallize, Filter and Desolventize Shea Butter

TIME/DATE STARTED	OUT OF	CONTAINER I.D.	CONT #/#	CONT TYPE	NET WT (kg)	INIT
	BC2	BC2/h. out/shea butter solids	1/2	MD	~~108.0~~	
	"	BC2/l. out/shea butter miscella	1/1	MJ4	45.0	
					153.0	
	MP3	MP3/K.O. pot/Hexane & water	1/1	PP	4.3	
	RC2	RC2/h. out/shea butter solids	2/2	MD	~~5.2~~	
					162.5	
dept 0100 13.14	F1	F1/OUT/SHEA STERINE FRACTION	2/2	PP	16 Kgs	×2
"	"	"	1/2	PP	~~14.6~~	
13:45	"	F1/BLOW/SHEA STERINE FRACTION	1/1	PP	5.7	
1545	"	F1/Frame/Shea Cake	1/1	6	4.0	
1515	"	F1/Can/Shea Line drain	1/1	6	2.4	
1850	MP3	MP3/OUT/Shea olein Fraction	1/2	PP	16.1	
		" " "	1/2	PP	5.6	
					21.4	
PAGE TOTAL						

Containers: Use Equipment I.D. or the following:

BS-Bulk Sack	G-Garbage	MP-Metal Pail	PS-Paper Sack	CS-Cotton Sack	MD- Metal Drum
LB-Large Bucket	PD-Plastic Drum	SC-Sample Container	FD-Fiber Drum	PT-Tote	PP-Plastic Pail
					PB-Porta Bin

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 7 of 9

PROCESS DATA – MR7

Page 1 of 3

TIME (every hour and change)		Target	00:15	01:20	02:20	3:20	4:20
DATE	Month/ day		Aug 31/00	N/C	N/C	N/C	N/C
Operator	Init		GS	GS	GS	GS	GS
MR7 Volume	L	Var	N/A	N/A	N/A	N/A	N/A
MR7 temperature	°C	5	37.4	14.1	12.5	10.8	7.0-9.8
MR7 agitator speed	RPM		9.5	9.5	9.5	9.5	11
MR7 glycol set temp.	°C	3	3	3	3	-5	-5
MR7 glycol flow	Lpm	Var	N/A	N/A	N/A	N/A	N/A

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 7 of 9

PROCESS DATA – MR7

Page 2 of 3

TIME (every hour and change)		Target	5:30	6:25	0750	0850	0950
DATE	Month/ day		Aug31/00	N/C	"	"	"
Operator	Init		GS	GS	D.L	D.L	D.L
MR7 Volume	L	Var	N/A	N/A	NA	NA	NA
MR7 temperature	°C	5	3.9-7.0	4.6-6.7	4.3-	3.6-4.5	3.8-4.6
MR7 agitator speed	RPM		11	11	11	11	11
MR7 glycol set temp.	°C	3	3	3	3	4	4
MR7 glycol flow	Lpm	Var	N/A	N/A	NA	NA	NA

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 7 of 9

PROCESS DATA – MR7

Page 3 of 3

TIME **(every hour and change)**		**Target**	1055	1155	13:30	15:05	
DATE	Month/ day		Aug/31/00	NC	NC	NC	
Operator	Init		DA	DA	DA	RN	
MR7 Volume	L	**Var**	NA	NA	NA	NA	
MR7 temperature	°C	**5**	3.7-4.7	4.1-4.7	6.2	6.3	
MR7 agitator speed	RPM		11	11	11	11	
MR7 glycol set temp.	°C	**3**	4	4	4	4	
MR7 glycol flow	Lpm	**Var**	NA	NA	NA	NA	

PROCESS DIRECTIVE

Project No.: 300-99856
Process Leader: Herve Douce
Process Technician: Greg Gervais

Aug31-Sept 1, 2000

PROCESS: Crystallize, Filter and Desolventize Shea Butter

PROCESS DATA - Centrifuge

Page 1 of 1

TIME **(every change and every hour)**		1610				
Operator	init	GG				
Max fill weight	Kg	40				
Min fill weight	Kg	30				
Load/Dewater speed	Rpm	750				
Dewater time	Sec	45				
Spin time	Sec	360				
Spin speed	Rpm	1900				
Discharge speed	Rpm	500				
Oxygen level	%	1				
Bearing housing nitrogen	Cfm	0				
Labrynth nitrogen	Cfm	2.3				
Nitrogen inlet pressure	psi	42				
Discharge weight	Kg	17				
Discharge solids (wet, dry, etc.)		damp				

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais
PROCESS: Crystallize, Filter and Desolventize Shea Butter

PROCESS SAMPLES

SAMPLE I.D.	NUMBERxSIZE	DETAILS	TIMES/INIT
MP3/out/desolventized shea stearine	1 X 100ml	Take to lab for analysis.	0815
F1/out/Shea stearine fraction	2 x 250ml	Take while pailing.	13:10
MP3/out/desolventized shea miscella	1 x 100ml	~~Take to lab for analysis~~ TAKE AFTER 3 hr hold.	1810
MP3/out/Shea oleine fraction	2 x 250 ml	Take while pailing.	1809

PROCESS DIRECTIVE

Project No.: 300-99856 Aug31-Sept 1, 2000
Process Leader: Herve Douce
Process Technician: Greg Gervais S
PROCESS: Crystallize, Filter and Desolventize Shea Butter Page 6 of 9

Deviations: **Page** 1 1

Step #	INITIAL	Date	Deviation(s)
4.0	GL	Aug 30	Used 65psi valve
42.0	AJ	Aug 31	Extend cold to tomorrow morning.

Appendix 4B-1: Solving the darkening of the oil at start up

Trial #1: 2/22/99
Oil used: RBD canola oil (POS Startup oil)
Parameters :
Oil flow: 40% of rotometer scale
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Initial vacuum was 0.3 mm Hg.
The Spirafilm was set at 268°C with a variation between 265-271°C .
The column was set at 265°C with a variation between 263-266°C .
Spirafilm heater and column were heated to temperature **before** initiating oil flow to the column. After all the oil had passed over the column the deodorizer pot was heated to 265°C and held there for 20 minutes, the oil was then cooled to 100°C and the final product sample was taken. The first oil over the column was diverted to the drain pot. This oil was dark in colour. The final product was lighter in colour but was still darker than the starting oil. The deodorization process was darkening the oil.
Note: The cool down procedure of the Spirafilm heater and column was initiated once **all** the oil had passed over the column.

	Sample		
	Starting oil	Drain Pot oil	Final Product
Peroxide Value	0.8	0.4	0.2
% Free Fatty Acids	0.06	0.03	0.03

From the above data it appears that the increase in colour was not due to oxidation of the oil or free fatty acids. Oil colour may increase due to polymerization, oxidation, thermal degradation of phosphorus, or condensed fatty acids falling into the deodorized oil (vacuum loss). During this run the vacuum was good, therefore oxidation and fatty acid condensation should not have occurred. The oil had no phosphorus. Polymerization could have occurred just before cool down as the flow rate of oil over the Spirafilm and column would be tapering out at that time.

Trial 2: 2/24/99

Oil used: RB Palm oil
Parameters
Oil Flow rate: 40% of rotometer scale, i.e. Oil Rate of 2.27 Kg/hr.
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Hold deodorizer pot at 100°C until feed is finished then heat to 265°C and hold 20 minutes, then cool to 100°C and discharge.
Collect start up oil in drain pot until column temperature is at 260°C .
Note this oil had no phosphorus.
The Spirafilm was set at 269°C with a variation between 265-288°C .
The column was set at 265°C with a variation between 264-266°C .
Time to heat ~ 2900g of oil from 100-265°C was 55 minutes (3°C /minute).
The diverted drain pot start up oil was dark.

	Sample		
	RB Palm oil	Drain Pot oil	Final Product
Peroxide Value	0.00	-	0.00
% Free Fatty Acids	0.25	-	0.02
Colour 5.25" Lovibond [a]	70.0Y 18.1R	dark	34.0Y 3.0R

[a] The Lovibond scale was used because the oil was too dark for the AOCS scale.

It should be noted that as in the previous run (2/22/99) this oil had no phosphorus, the vacuum was good and the oil's temperature was kept below 300°C .
The dark start up oil was analyzed for polymerized and oxidized triglycerides, it was found to contain a 6.44% polar fraction (the usual value is 2%). The polar fraction contained 5.25% polymerized triglycerides and 17.17% oxidized triglycerides, therefore the dark colour was due to polymerization. Polymerization can occur at temperatures of 150°C and higher. During start up the column was heated to 265°C with no oil flow, the column was also cooled with no oil flow; this provided ample opportunity for the thin film of oil on the column to polymerize.

The start up and cool down was modified so that oil was flowing over the Spirafilm heater and the column during start up and cool down. The oil during start up and cool down was diverted to the drain pot as stripping of FFA would not be complete at lower temperature. When this procedure was followed we noted that the startup and cool down oil was no longer dark.

Trial #3: 3/3/99

Oil used: **Atmospheric bleached Shea butter**
Parameters
Flow: 40 % of rotometer scale
Deodorization temperature: 265°C
Sparging gas: Nitrogen
Heating and cooling of the Spira heater and column was done with Shea butter flowing over these units.
Place ice and salt in the distillate trap to strip maximum amount of FFA before vapours reached the vacuum pump.
Hold deodorizer pot at 100°C until feed is finished then heat to 265°C and hold 2 hours. Cool to 100°C and discharge.
Collect start up oil in drain pot until column temperature is 260°C .
Note that this oil had no phosphorus.

Sample	% Free Fatty Acids	AOCS 5.25" Colour
SM/bleached Shea	2.03	9.9Y 0.6R
Drain Pot column at175°C	-	9.9Y 0.9R
Drain Pot column at 250°C	-	9.4Y 1.5R
Drain Pot column at 250°C	-	9.4Y 1.3R
Deodorizer at 265°C Time: 0	0.26	9.0Y 1.1R
Deodorizer at 265°C Time: 20 Min	0.19	9.0Y 1.2R
Deodorizer at 265°C Time: 60 Min	0.15	9.9Y 1.2R
Deodorizer at 265°C Time: 120 Min	0.05	8.5Y 1.1R

This trial demonstrated that the deodorizer can remove 97.5% of the Free Fatty Acids and that heat bleaching can reduce the yellow colour by 1.0 unit. It was also noticed that the red colour rose from 0.6 to 1.5; through heat bleaching this was reduced to 1.1.

The decrease in red seemed to occur when the temperature was around 265°C . It seems that some thermal degradation product in the atmospheric bleached Shea increased the red and this was partially removed during heat bleaching. The same behavior was observed when atmospheric bleached Shea was deodorized in a glass deodorizer.

Trial #4: 3/4/99

Oil used: **Atmospheric bleached Shea butter**
Parameters
Oil flow rate: 40% of rotometer scale.
Deodorization temperature: 265°C
Stripping gas: 3% steam
Note: Burkina Faso Clay was used instead on tonsil 120FF

Heat and cool the column with Shea butter.
When column temperature is less than 240°C collect oil in the drain pot. Place ice and salt in the distillate trap. Hold deodorizer at 100°C until feed is finished then heat to 265°C and hold 2 hours, then cool to 100°C and discharge. Collect oil in drain pot until column temperature is 260°C . Note that this oil had no phosphorus.

Losses during this run to heating and cooling : 870g, the deaerator was loaded with 2530.8g of Shea butter.

Sample	% Free Fatty Acids	AOCS 5.25" Colour
SM/BK. bleached Shea	1.70	9.9Y 0.8R
Deodorizer at 265°C Time: 0	0.02	10.0Y 1.4R
Deodorizer at 265°C Time: 1 hr	0.02	9.4Y 1.3R
Deodorizer at 265°C Time: 2 hr	-	9.7Y 1.4R

Once again deodorization increased the red. Heat bleaching did not occur for the red colour and only slightly for the yellow. The above Table shows that if oil passing over the column at temperatures below 240°C is discarded (start up and cool down oil) then a holding period in the deodorizer at 265°C is not necessary (this saves a minimum of 1.5 hours of operation time).

Trial #5: 3/5/99

Oil used: **Atmospheric bleached/Carbon Treated High FFA Shea butter.**
Initial colour of oil was very dark.
Note: This oil was first bleached with 2% clay , then it was treated with 0.5% carbon
Parameters
Oil flow rate: 60% of rotometer scale
Deodorization temperature: 265°C
Stripping gas: Nitrogen
Heat and cool the column with Shea butter.
When column temperature is less than 220°C **collect** oil in the drain pot. Place ice and salt in the distillate trap. Heat deodorizer to 265°C when feed is started, hold 2.5 hours at temperature after feed to the deodorizer has stopped, then cool to 100°C and discharge.

Sample	**% Free Fatty Acids**
SM/bleached High FFA Shea butter	13.14
Drain Pot - Column at 247°C	1.80
Deodorizer 240°C - Time : 0	0.28
Deodorizer 1 hour at 265°C	0.18
Deodorizer 1.5 hours at 265°C	0.17
Deodorizer 2.0 hours at 265°C	0.12
Deodorizer 2.5 hours at 265°C	0.08
Drain Pot -- Condensate from column during batch deodorization	0.12

During this run the distillate trap began to plug at the vapor inlet to the distillate trap. The plugging problem can be overcome by keeping the ice in the trap 12 cm below the top of the trap, also a heating tape could be placed around the top. In future runs the deodorization temperature should be kept at 100°C , if the deodorization temperature is above 180°C and vacuum is lost then fatty acids that have been distilled from oil in the deodorizer may fall back into the oil and affect the final colour. In the event of vacuum loss the flow from the column can be diverted to the drain pot, this should prevent unrefined oil from the column from falling into the deodorizer. The above procedure will increase operation time but should produce a better product.

It Is noted from the above Table that the column was able to remove 86.30% of the FFA present at a flow rate of 60% of rotometer scale, perhaps a slower flow rate would remove more.

Appendix 5A-1: Dependence of overall yield on starting Shea specifications

Processing Step	Assumptions	Theoretical Yield of low quality Shea Butter	Theoretical Yield of high quality Shea Butter
Bleaching	6% clay & 1% carbon dosage with low quality Shea Butter	88% *	-
	1.5% clay dosage with high quality Shea Butter	-	96% *
Physical Refining	6.52% of free fatty acid in low quality Shea Butter.	89.0% **	-
	2.0% max. free fatty acid in high quality Shea Butter.	-	93.5%**
Overall Yield Ψ		**78.3%**	**89.8%**

Ψ See Appendix 4B-2 for calculation.
* 30% by weight of entrained neutral oil in weight of bleaching clay used. See Appendix 4B-2.
** 1.0 % of neutral oil entrainment in deodorization. See Appendix 4B-2.

APPENDIX 5A-2 : Calculation of yields

Yield after bleaching:

With low quality Shea butter batch used in the pilot plant we use 6% clay dosage.

Yield after bleach = $\frac{\text{Wt of bleached Shea butter obtained in plant}}{\text{Wt of SM/Shea butter used at start of bleach}}$

$= \frac{819.00}{944.83} = 88.0\%$

If colour of SM/Shea butter was brought under control by proper processing in country of origin it is assumed that the amount of clay needed will be around 1.5% and that there will be no need for activated carbon.

Assuming 30% by weight of entrained neutral oil in weight of bleaching clay used and assuming a 20 kg filter press drain lost.

Yield, based on 1 tonne of SM/Shea after bleach will then be

$$= \frac{(1000-(0.3 \times (1000 \times 0.015))-20}{1000} = 96\%$$

Physical Refining Yield:

With a low quality Shea Butter the % of free fatty acid in the Bleached oil was 6.52%. Assume 1% neutral oil loss in deodorizer and 35 kg of transition oil at beginning of deodorization.

Yield, based on one tonne of SM/Shea after deodorization :

$$\frac{\text{Wt of deodorized oil}}{\text{Wt of bleached oil}} = \frac{\text{Wt of bleached oil - Wt of FFA- 1\% N. oil loss}}{\text{Wt of bleached oil}}$$

$$= \frac{1000 - 65.2 - 10.0-35.0}{1000} = 89.0\%$$

If the Shea seed were properly collected and stored a maximum free fatty acid content of 2.0% in the crude oil can be assumed.

Then the yield after deodorization will be: $\frac{1000 - 20.0 - 10-35.0}{1000} = 93.5\%$

OVERALL YIELD:

Overall yield for low quality Shea Butter:

88% x 89%= 78.3%

Theoretical Overall yield for Shea Butter with low colour and FFA, (2%):

96% x 93.5%= 89.8%

Thus if the starting material is top quality Shea butter approximately (898-783)= 115 kg of additional bleached and deodorized Shea butter per tonne processed will be achieved.

APPENDIX 5A-3. Calculation of costs at POS

A: Using low quality starting Shea butter

Estimated processing cost for refining SM/Shea butter with ~ 6% Free fatty acid content and dark colour, AOCS 20.0Y3.7R using 1" cell. An overall recovery of 78.3% has been assumed.

Amount of SM/Shea butter processed	1 Tonne	2 Tonnes	4 Tonnes
Labour at POS	$5,730.00	$6,600.00	$8,725.00
Material	$1,580.00*	$2,300.00	$3,300.00
Equipment	$3,300.00	$3,975.00	$7,850.00
Lab Supplies	$40.00	$40.00	$40.00
Total	$10,650.00	$12,915.00	$19,915.00
Processing cost per kilo of SM/Shea butter	$10.65	$6.46	$4.98
Processing cost per kilo of final RB Shea butter	$13.60	$8.25	$6.36

* See appendix 4B4 for estimation of material cost for processing 1 tonne of Shea butter.

High quality starting Shea butter.

Estimated processing cost for refining SM/Shea butter with ~ 2% Free fatty acid content and light colour, AOCS 70.0Y2.9R using 5 1/4" cell:
An overall recovery of 90% has been assumed.

Amount of SM/Shea butter processed	1 Tonne	2 Tonnes	4 Tonnes
Labour at POS	$5,730.00	$6,600.00	$8,725.00
Material	$1,450.00	$1,900.00	$2,800.00
Equipment	$3,300.00	$3,975.00	$7,850.00
Lab Supplies	$40.00	$40.00	$40.00
Total	$10,520.00	$12,515.00	$19,415.00
Processing cost per kilo of SM/Shea butter	$10.52	$6.25	$4.85
Processing cost per kilo of final RB Shea butter	$11.69	$6.95	$5.39

Appendix 5A4. Material usage for processing 1 tonne of low quality Shea butter

Material	Quantity used	Total Cost, $ Can.
Citric acid	1.88 kg	5.45
Bleaching Clay	56.68 kg	85.02
Activated Carbon	9.40 kg	57.37
Nitrogen		34.50
Filter aid	0.57 kg	0.74
Canola oil	170 kg	945.00
Pails & lids	46	257.70
Filter papers	16	38.40
Cartridge filters	2	56.00
Waste Disposal		50.00
Total		**$1,530.18**