Carmine Dye Extraction Process and the Cochineal Insect

Introduction

Peru is the major supplier of carmine dye, a natural, red colouring agent derived from the cochineal insect. The dye is used in foods, drugs, and cosmetics. As a result of global restrictions on artificial colorants in food and other consumer items (many synthetic red dyes are now prohibited in the United States), Peru enjoys a considerable advantage in the world market, supplying 80% of the world's cochineal — about 40% as a dye and 60% in insect form.

The Peruvian government wants to increase the processing of carmine dye in Peru, given that cochineal insects are plentiful, rural people are experienced in harvesting the insects from the prickly pear cactus, and extraction techniques are comparatively simple. By locating processing plants close to cochineal production areas, rural industry and local employment can be encouraged. Currently cochineal "farmers" only earn an estimated 10% of the revenue generated from cochineal processing. An estimated 50 000 people harvest the insects by hand, dry them in the sun, and sell them through intermediaries to carmine processors in Lima.

Peru's Instituto de Investigacion Tecnologica Industrial y de Normas Tecnicas (ITINTEC), and the Simon Fraser University have worked together to improve the carmine dye extraction process, providing a 23% yield of 62% pure carmine. (Other commercial processes result in 20-23% yields of 52% pure carmine.) A pilot production plant with a capacity to produce 5 kg of carmine per day has been constructed.Extracting carmine powder from the insects involves boiling the insects in water, followed by filtration, precipitation, and washing and drying the final product.

Prerequisites

Access to cochineal and markets for the red dye. Cochineal grow on prickly pear cacti in the Andean region and in general in very dry regions.

Potential users

Cochineal exporters and producers as well as current producers of carmine interested in improving yield.

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Resources

In the IDRC library:

Simon Fraser University, Burnaby, BC. 1992. *Final technical report / Carmine Production from Cochineal (Peru) II*   s.l. : s.n. 1 v. in various pagings : ill. PROJECT NO: 87-1021   (Final report)   lan: E   date: 1992   isn:94238   statc: RCMPL   astat: COMPL   location: ARCHIV 547.975(85)   S 5   MFICHE IDRC-Lib-94238

L'extraction du carmin de cochenille

Introduction

Le Pérou est le principal fournisseur de carmin, colorant rouge naturel tiré de la cochenille qui sert en alimentation et entre dans la composition des médicaments et des produits de beauté. Étant donné les restrictions mondiales imposées sur l'utilisation de colorants artificiels dans les aliments et d'autres produits de consommation (beaucoup de colorants rouges synthétiques sont maintenant interdits aux États-Unis), le Pérou a un avantage considérable sur le marché international, auquel il fournit 80 % de la cochenille (environ 40 % sous forme de colorant et 60 % sous forme d'insectes).

Le gouvernement péruvien veut augmenter le traitement du carmin au Pérou car la cochenille y abonde, les ruraux sont passés maîtres dans l'art de récolter les insectes qui nichent dans les figuiers de Barbarie et les techniques d'extraction sont relativement simples. L'installation d'usines de transformation à proximité des zones de production de cochenille peut favoriser l'essor de l'industrie rurale et la création d'emplois à l'échelle locale. Les producteurs ne touchent présentement que 10 % environ des revenus provenant de la transformation de la cochenille. Les données estimatives établissent à 50 000 le nombre de personnes qui prennent part à l'opération : les insectes sont cueillis à la main et séchés au soleil avant d'être vendus par des intermédiaires aux transformateurs de carmin à Lima.

En collaboration avec l'Université Simon Fraser, l'Instituto de Investigación Tecnológica Industrial y de Normas Técnicas (ITINTEC) du Pérou a cherché à améliorer le procédé d'extraction du carmin afin d'obtenir un rendement de 23 % de carmin pur à 62 %. (D'autres procédés commerciaux donnent un rendement de 20 % à 23 % de carmin pur à 52 %.) On a construit une usine pilote capable de produire 5 kg de carmin par jour. L'extraction de la poudre de carmin se fait en plongeant dans l'eau bouillante les femelles desséchées de l'insecte, pour ensuite filtrer le résidu avant de passer à la précipitation, au lavage et au séchage du produit final.

Préalables

Accès à la cochenille et débouchés pour le colorant rouge. La cochenille se reproduit sur les figuiers de Barbarie des pays andins et, en général, dans les régions désertiques.

Utilisateurs éventuels

Exportateurs et producteurs de cochenilles ainsi que les producteurs actuels de carmin intéressés à améliorer leur rendement.

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Carmine Extraction Technology

IDRC Carmine Extraction Process

During 1989 - 1990, IDRC sponsored collaborative work between ITINTEC (Peru) and Simon Fraser University (B. C.) to develop a process flow sheet and pilot plant for the extraction of carmine, the complex Al / Ca chelate of carminic acid, from dried cochineal. The extraction process so developed has been shown to yield 70- 80% of the carminic acid contained in the dried cochineal feedstock, producing an aluminium/calcium lake salt (carmine) containing 65% by weight carminic acid. This was considered a premium product compared to the then existing industry standard of 52 - 55% contained carminic acid in commercial carmine. These results were subsequently confirmed 1991- 1993 in further IDRC sponsored work performed by CODETEC (Brazil), who extended the scope of the R&D somewhat by conducting bench-scale tests on the extraction of carminic acid and a water soluble variant of carmine lake. Both organisations, ITINTEC and CODETEC, generated business projections for a green-field plant rated at ~1000 kg/ month carmine production capacity. Of the overall project activity, only the portion related to carmine lake powder production is discussed here. The IDRC carmine extraction process follows:

The first step is to **SIEVE** the dried # 1 grade cochineal to remove the finer particulate material (lower carminic acid content). Only material retained on a #10 - #14 sieve is processed, with the reject material being set aside for modified secondary treatment. The coarse fraction of the retained raw material is then **DEGREASED** with an organic solvent (e.g., hexane) to remove waxes and grease from dried body parts, and the cleaned and dried feedstock is then **MILLED** to -100 mesh sieve size to break down the tissues containing the colorant, and so accelerate the **EXTRACTION** phase and maximise carminic acid dissolution and recovery. The ground cochineal is fed into a stainless steel reactor containing boiling alkaline water, i.e., a solution of sodium carbonate at pH 9, steam heated to 95-100 C, using a batch loading rate of 20-25 gm cochineal / litre. Vigorous mechanical stirring of the closed reactor contents during the entire 15 to 30 min. treatment period is critical to ensure maximum extraction of the carminic acid. Recovery levels range from 70 to 80 % of the carminic acid in the feedstock. The boiled carminic acid solution is then filtered in situ by adding a flocculent to help reduce turbidity by passing or pressing the alkaline liquor through a 120 mesh sieve to retain the solid cochineal residue.

PRECIPITATION of carmine from the hot solution is then achieved by reducing the pH to 5.5 - 5.0 by adding citric acid; simultaneously adding appropriate amounts of aluminium (alum) and calcium (chloride) salts; boiling the treated liquor at 100 C for 15-20 min., during which time the Al /Ca salt of carminic acid precipitates from solution. Next, **SEDIMENTATION** of the carmine occurs by gravity over a period of 1-2 hours, following which the mother liquor is drained and the wet sediment transferred to a separate unit for disc **CENTRIFUGATION**. The time dependent stages, from extraction through centrifugation, dictate batch cycle times for these steps in the range of 4-6 hours, depending on the volume of throughput.

The resultant carmine deposit is cleaned by **WASHING** with de-ionised water to remove any soluble impurities carried over from the reactor. The washed product is **STERILISED** at 120 C, then put through a **DRYING** process at 40-70 C under partial vacuum to ensure a low residual moisture content (< 3%) in the final product. The carmine may be **MILLED** in a stainless steel ball mill to suit client specifications on final particle size distribution. Finally, the carmine powder is tightly sealed in the **PACKAGING** line in 10-25 kg moisture-proof polythene containers ready for shipping.

Quality and process control require

: carminic acid and trace element analysis of the feed material and the carmine product;
: end-point monitoring of the extraction stage ;
: pH control throughout ; and
: colour certification of the product.

No procedures have been identified for the secondary processing or disposal of solid and liquid waste streams.

To achieve these process steps, the following partial list of equipment will be required, scaled of course, in size and capacity to suit the intended production rate of carmine, say, about 10- 40 kg carmine per day (24 hrs). The list below shows the size or capacity of the item envisaged for a batch size of the order of 50 kg dried cochineal that will yield 11-12 kg carmine.

# Units	Unit Description	Capacity/Size/Rating
1	stainless steel extraction tank with stirrer	2000 litres/ 3 HP stirrer @ 30 rpm
1	stainless filter basket	120 litres
1	stainless steel precipitation tank	4500 litres/ 3 HP stirrer @ 100 rpm
5	stainless steel storage vessels	4500 litres, 2 with lid-mounted sieves
1	stainless steel ball mill	10 kg / hour
1	hammer mill	15 kg / hour
1	disc centrifuge	50 litres
1	tunnel drier	1 m dia. x 2.5 m length
1	boiler	40 HP, with water softening
1	sieving machine	(8 - 150 mesh)
2	weigh scales	Up to 150 kg
1	electronic balance	Up to 2 kg
1	de-ionised water supply system	50 litres / min.
1	packaging machine	

To effect proper quality control of the process and product, a laboratory test facility will be required, containing :-

colorimeter	pH meters

spectrophotometer	wet chemistry equipment
analytical balance	particle size analyser

The market identifies 2 distinct grades of carmine, one for the food industry, with a blueish hue to the red colour; and the second for cosmetic applications, distinguished by its yellow tone. These colour differences are quantitatively defined, and assessed, by the colorimeter. Other product characteristics controlled to suit customer specifications include trace element analysis (e.g., Pb, As), carminic acid content, and particle size distribution.

Overview of the Cochineal / Carmine Industry

On the supply side of the equation, Peru dominates the production volume and pricing of both the raw material, cochineal, and its processed carmine products. At the user end of the market chain, the US and European food, pharmaceutical , and cosmetics manufacturers look for the same constancy of price and quality in carmine that they were used to with synthetic colorants, and tend to look to Western suppliers, rather than Peru , to guarantee this consistency and repeatability of product in terms of colour and purity. The carmine industry therefore has a primary component extracting carminic acid products from cochineal, and a secondary sector which buys, blends, homogenises and packages carmine products for end user consumption.

Many Western companies only blend and package, while a smaller number bridge the supply line from purchased cochineal to processed and standardised carmine products ready for use in consumer goods. It is believed that, of the South American carmine producers, Peru, and to a lesser extent Chile, do not sell directly to the end user.

As noted above, Peru continues to dominate the industry, harvesting 80% of the world's production of dried cochineal. From that, Peru supplies 82% of the cochineal processed outside of South America, and itself makes 40% of the world's carmine products. Before exploring such statistics in more detail, however, it is useful to record some facts about the industry, starting with the beetle itself. It is estimated that approximately 80% of the annual cochineal harvest is collected from wild plants, and the balance from managed plantations. The latter are increasingly associated with on-site processing plants, e.g., Colca APX in Peru, and Colores de Chile in Chile. The median yield of dried cochineal from plantations is about 500 kg over 3 harvests per hectare per year, although yields may range from 300-1500 kg depending on plant size and the percentage of the plant surface infested. Following harvest, the drying process reduces beetle weight to 1/3 of original. The dried cochineal is graded # 1, 2, or 3 in order of decreasing particulate size, and , it so happens, carminic acid content. Grade #1 is the common export quality, containing 20-22 % carminic acid. The product is sold by the rural farmer / family to a collector, then to a wholesaler, and finally to the exporter. It is not known what fraction of the eventual sale price, e.g., USD 27/kg FOB Lima , is retained by the rural farmer who harvested the cochineal.

The dried cochineal is then processed in a batch system to yield carmine products. The processing stage is kept highly secret , indeed the only public disclosure is that made by ITINTEC on the details of the IDRC extraction work. Information on the products is more accessible. There are at least 4 identifiable forms of cochineal-based colorant. The most common is the carmine lake, as produced by IDRC's process. This is a complex aluminium / calcium salt of carminic acid, insoluble in water, with different grades containing between 50 and 65 % carminic acid.. It may be converted into a water and acid soluble chelate variant by further chemical processing. The third form of colorant is carminic acid, a red-orange powder produced by treating and spray drying the liquor from the initial alkaline digestion step of cochineal processing. This variant is therefore aluminium and / or calcium free, and is readily soluble in water and common organic solvents. Lastly there appears to be some considerable trade in carmine solutions, i.e., solutions containing specified concentrations (3-10 %) of carminic acid. Note that the 3% solution is sometimes referred to as ' cochineal '. The largest volume product of these four is carmine lake, containing a minimum 52 % carminic acid. This is derived from dried cochineal containing a nominal 20 % carminic acid, so that neglecting wastage, 2.5 kg dried cochineal are required to produce 1 kg of carmine at 50 % carminic acid content; or 3.25 kg cochineal yield 1 kg carmine containing 65 % carminic acid. Allowing for some wastage and less than perfect recovery, one may note that 3 to 4.5 kg cochineal are required to produce 1 kg carmine. Linking this to harvesting cochineal in an integrated plantation, 1 hectare may yield about 100 kg carmine per annum.

Turning now to the industry production statistics, it is regretted that only data for 1996 have been identified in sufficient detail, and shown below in Table 1.

Table 1. Global Production Survey: Cochineal

	Dry cochineal produced	Cochineal processed in country	Cochineal exported
Peru	410	180	230
Chile	40	30	10
Canaries	20	0	20
Bolivia	10	1	9
Central America	15	1	14
Developed countries	0	283	0
World Totals	495	495	283

Notes:

- Production figures are in metric tonne , based on 1996 data.
- Central American countries include Mexico, Honduras, El Salvador, etc.
- The developed countries importing and processing cochineal to carmine products include Spain, Germany, France, Japan, Italy, USA and the UK.

The uncertainties in the data are more significant with respect to Peru than any of the other producers. The figures of 410 MT harvested and 230 MT exported are probably low as a result of incomplete reporting by producers to government offices. Unofficial sources put the total cochineal production in Peru at > 500 MT in 1996, 750 MT in 1997, and > 750 MT in 1998, with approximately the same percentages of that harvest processed in Peru.

The steady historical growth of cochineal and carmine production in the period 1985 to 1995 was completely upset in 1995 when the European regulatory aspects of food colouring were changed in favour of the use of natural colorants. Suddenly in 1995, the demand for cochineal exceeded supply by some 100 MT. Prices increased steeply as commodity speculation kicked in. At the higher prices, more insects were harvested than ought to have been, creating a perceived future shortage, and further driving up the price. The market settled down again in 1998, with supply and demand in balance, and prices at normal industry levels (see below). The lesson illustrated is perhaps that the market is, by its structure, potentially volatile, and not a secure place to invest in processing technology.

Data were obtained on the extent of global activity in 1996 on cochineal processing, and the results are given below in Table 2. Where specific data on product variants were unobtainable, it was assumed that the product mix made and sold by Peru represented the global distribution of demand for carmine variants, and the activity per variant was calculated accordingly. Thus in the row for Western processors - ' Developed countries ' - the assumption is that the 283 MT cochineal processed was distributed between lake, acid, and solutions in the same proportions as the documented distribution of processed cochineal in Peru. The self consistency of the data in Tables 1 and 2 is reflected in Note 3 in Table 2.

Table 2. Global Production Survey - Carmine Products (metric tonne)

	Carmine Lake	Carmine Acid	Carmine Solutions	Totals
Peru	50.0	8.5	27.0	N/A
Chile	8.3	1.4	4.0	N/A
Bolivia	0.3	0.05	0.15	N/A
Central Americas	0.3	0.05	0.15	N/A
Developed countries	78.6	13.4	42.5	N/A
World Totals	137.5	23.4	73.8	N/A
Carminic acid content	71.5	23.4	7.4	102.3

Notes:

- It was assumed that the Peruvian product mix for 1996 reflects the world demand for carmine products.
- Carminic acid content of lake and solutions are taken as 50% and 10% respectively.
- 102.3 MT of carminic acid from global consumption of 497 MT of dry cochineal reflects recovery of a 20.7% carminic acid content in the cochineal.

To illustrate the inherent consistency of Peruvian data for 1996, a mass balance of carminic acid was performed and is shown in Table 3. Column B in that Table repeats the published statistics for production, presumably entirely for export as lake, acid or solution, and col. D derives the carminic acid equivalent involved, and hence the cochineal feedstock used. The derived export figure for cochineal is seen to be very close to that separately published.

Table 3. Cochineal - Carmine Mass Balance - Peru 1996

A Processed Product	**B** Production tonnage	**C** Carminic acid content	**D** Contained carminic acid	**E** Cochineal processed
	MT	%	MT	MT
Carmine lake	50	50	25	125
Carminic acid	8.5	100	8.5	42
Solutions	27	10	2.7	13.5
Total cochineal processed in Peru				180.5
Total cochineal harvest in Peru				410.0
Total cochineal exported from Peru				229.5

Notes:

- Dry cochineal contains 20% carminic acid. Thus col. E = 5xD assuming 100% extraction efficiency.
- Carmine solutions may contain 7.5 to 15% carminic acid. A nominal level of 10% is used here.
- Reported tonnage of #1 grade cochineal exports is 231MT (c.f. 229.5 above).

Finally the supply/demand imbalance and the speculation in 1995-1996 are illustrated in the price scenario documented in Table 4, where it is seen that in a very short period of time, the FOB price for cochineal increased by a factor of 3 to 4, and the multiple for carmine was 2 to 3. The modest level of 1998 figures reflects that the Peruvian supply capacity for cochineal, at 750MT per annum, is now in balance with demand. The Peruvian supply side is critical because:

a) There is as yet no national sales point for the raw and processed products, allowing foreign buyers to exploit the competition between producers.

b) The cochineal and carmine sectors in Peru are by and large separate entities in a business and ownership sense, so that the carmine producers must to a degree compete with the world market to buy cochineal.

c) It is quite possible that if the price of cochineal drops below present levels, the local carmine producers will buy and warehouse a high percentage of Peru's cochineal production, creating an artificial scarcity of cochineal for foreign processors, and so leading to a new round of price increases for the raw commodity, and , of course, for the carmine derivative.

Table 4. Market Price Survey
(Price range in USD / kg)

	1990	1994	1996	1998
Cochineal	10 - 15	20 - 35	60 - 80	25 - 30
Carmine	100 - 120	120 - 150	250 - 370	130 - 150

Notes:-

- 1 kg of carmine lake @ 50% carminic acid requires input of 3 - 4 kg dry cochineal. Thus the market price of carmine must always be > 4 times the input cost of cochineal to properly accommodate processing value added.

- Public data sources were tapped for prices 1990-1996. The figures shown for 1998 were obtained from two separate unofficial sources.

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Relevant Web Pages

<u>Producers</u>

Natural food colours	www.aarkay.com/product
Warner Jenkinson Europe	www.colourcreators.com

Colores de Chile	www.cochineal.cl
Lansur	www.misti.lared.net.pe
Propagacion de cochinilla	http:// ekekop.rcp.net.pe
Global Natur	www.globalnatur.es
Saporiti	www.saporiti.com.ar/colorantes
Canary cochineal	www.arrakis.es/~rpdeblas/cochinea
Colca APX - Grupa Inca	www.colca.com

Health etc.

Allergy reaction	www.med.umich.edu/opm/newspage/dye
FDA - color additives in food and drugs	www.cfsan.fda.gov
BIBRA toxicity profiles	www.bibra.co.uk
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