SUMMARY

The wax is a God gift and is as old as man. The term wax is Anglo-Saxon Weax and was applied to the natural material of honey comb of the bee. Subsequent similar material were also known as Weax or Wachs, and later called as Wax. Waxes of commercial importance are of animal, mineral or vegetable origin. Amongst the animal waxes are the bees wax, wool wax and the Chinese insect wax. Montan, Ozokerite and Paraffin waxes have mineral origin. The vegetable group is represented by carnauba, ouricuri, candelilla, Japan and sugar-cane wax. Certain synthetic compounds which are not waxes from the standpoint of chemical composition, but do have waxy physical characteristics, are included because of their value in technical use as wax substitutes. Besides other natural waxes, some synthetic waxes have also come in the market. But still the best form of wax is natural wax found coated on vegetable plant products such as Carnauba, Ouricuri, Candelilla etc.

In the chemistry of the components of the animal and vegetable waxes, the higher aliphatic hydrocarbons are the foundation stones upon which the chemical structure of the wax alcohols, wax acids and wax alkyl esters is built. The same hydrocarbons are the principal constituents of the paraffin waxes.

The cane sugar industry has several by-products of immense potential value, and press mud is one of them having high potential for wax. The recovery of wax from press mud
will save much of the foreign exchange i.e. to a tune of Rs. 6-7 crores. This wax which is present as a protective layer on the cane, gets mostly extracted into the raw juice during milling. The raw cane juice is subjected to a clarification process so as to get rid of impurities and colours for better sugar crystal production. During the clarification process, it precipitates along with other non-sugars. The precipitate after filtration, is called Press Mud which contains waxes. The percentage of wax (8-15%) in the sulphitation press mud is much higher than in the carbonation mud (1% approx.) and makes recovery a possibility from the sulphitation press mud. The wax from press mud is extracted by solvent extraction, by standard patented processes such as the Merz Process, and the De Smet Process. Wax thus produced, is crude sugar-cane wax. This wax is in the crude state. It is brownish black in colour, soft in nature and has high ash, and resin content, and has an unpleasant odour. It also suffers from other drawbacks like stickiness and presence of mineral matter. The crude wax has poor solvent take up and retention, and so has little industrial application. The crude wax has to be refined or modified so as to make it fit for industrial utility.

Standard Process of Crude Wax Extraction

The press mud is dried by suitable means to reduce the moisture content 3-4%. The dried press mud is then subjected to a solvent extraction process either of a batch type or continuous
type similar to the solvent extraction of oil from oil cakes. Solvents generally used are (i) n-hexane, (ii) n-heptane, (iii) solvent oil, B.P. 90-120°C or (iv) mineral terpentine, B.P. 135-190°C.

**Brief Review**

Attempts, by number of workers in India and abroad, have been made to refine the crude wax and further modify for different uses by using drastic chemicals and heat treatment etc. Kapil and Mukherjee reported that the inorganic constituents are eliminated from crude wax by heating with hydrochloric acid and subsequently deashed wax is defatted by the use of solvents like alcohol, acetone and petroleum ether. Wiggins bleached the wax by the use of potassium dichromate and sulphuric acid. Purification of cane wax by distillation under vacuum was next attempted by Hatt. Warth prepared synthetic wax by condensation of dibasic acids with glycols, known as synthetic I.G. Waxes.

The refining of crude wax as has been developed and reported elsewhere so far, consists of first deashing with conc. hydrochloric acid then defatting it using suitable solvents such as alcohol, acetone, etc., and subsequently bleaching it by chromic or chloric acids according to requirements. The bleached and refined wax is further modified by partial esterification and rest by ammination and then they are blended to requirement. In all the manufacturing processes of modification and refining the wax,
as tried at many levels, could not produce the requisite quality of wax as that of Carnauba, Candelilla or Ouricuri waxes and were not economically and technically viable.

The waxes so produced have been found to be of less industrial application because of their low solvent retention and jellying properties. These waxes when applied, used to crack apparently because of developed high resinous substances and low solvent retentivity.

Even at present our country is entirely dependent upon the foreign sources for the supply of about 1200 Tonnes of hard plant waxes like that of carnauba, candelilla, and other vegetable waxes which are imported in India costing foreign exchange to a tune of Rs. 6-7 crores per annum. This shows that a considerable amount of foreign exchange can be saved if Sugar-cane Wax of good quality, comparable to the imported waxes, is manufactured in the country.

The sugar-cane wax is an ester of higher fatty acids with monohydric alcohols such as ceryl and myricyl alcohols along with some free fatty acids, alcohols, sterols and hydrocarbons. Crude wax contains a fairly large proportion of palmitic, stearic, oleic, caproic and arachidic acids, all of which are readily saponifiable. The non-saponifiable material consists of about 80% of the higher alcohols, triacontanol or myricyl alcohol, about 10% of a mixture of sterols such as brassica -, stigma -, and sitosterols
and about 5% of pentatriacontane.

The crude cane wax, as obtained from press mud by any of the standard extraction systems, consists of oily and fatty matter (called herein as soft wax), hard wax and the resinous matter. In order to meet the trade requirement, it is necessary and essential to produce wax of high purity, a light colour and sufficient hardness.

Work done and Process Developed in the Study Presented

Under these circumstances, a process has been developed herein, utilizing a developed azeotropic mixture of solvents, employing the principle of synergism, for the manufacture of refined wax from crude cane wax obtained by either standard De Smet or Merz process.

In the previous processes, attempts were made by attacking and dissolving out the unwanted constituents of the raw material i.e. fatty matter, colour and inorganic substances by using drastic chemicals. Since the removal of these by chemical treatment were adversely affecting the product. Therefore, it was conceived that if on the other hand, the product itself is dissolved out by using suitable solvents, thereafter the solvents are evaporated out or distilled out, will result directly into a purer product and by-products.

Development of Solvent

With the above considerations, it was further conceived
that a single solvent may not be suitable for such fractionation of hard and soft waxes from crude cane wax. Therefore, it is desirable that a mixture of two solvents under the phenomenon of Principle of Synergism, if developed, will result into a suitable azeotropic (constant boiling) mixture which will be highly suitable for such a desired fractionation. Under the principle, this developed azeotropic mixture is supposed to have a lower boiling point than the average sum total of the two boiling points as well as will have a more efficient solubility and fractionation than the individual solvent.

As such the study involves the development of an azeotropic mixture and initially separating out by dissolving the soft wax at lower temp. treatment and hard wax at higher temp. treatment. The pitch is left behind and discarded. Further, the solvents, from the eluted mass of soft wax and solvent, and hard wax and solvent, are to be distilled out and reused such that the total quantity of desired solvent is drastically cut down, so as to make the process economically viable, which has been dealt in detail in the thesis.

From the efficiency of different available organic solvents it has been found that weakly polar hydroxylic solvents and weakly polar chloro-hydrocarbon solvents are the best suited solvents for fractionation of crude wax, since the crude wax also contains weakly polar hydroxylic and hydrocarbon based constituents.
The chloro hydrocarbon solvents have been selected because of the fact that a chloride group, in the solvent, bleaches the dark coloured components present in raw crude cane wax. Both types of solvents have been found efficient except that a few chloro-solvents also dissolve some of the hard wax at lower temperatures.

The study, on the phenomenon of mixed solvents for the development of a mixture of solvents which have a much higher solvent efficiency than the sum of their individual effects, has revealed that many of the combination of solvents are having the azeotropic properties. The azeotropic binary solvent mixtures i.e., an extraordinary constant boiling solvent mixture, have been prepared and the experiments conducted for fractionation of crude wax on lab. scale using selected azeotropic solvent mixtures, viz., isopropyl alcohol and symmetrical dichloroethane in the ratio of 1:3, has been found to be most efficient solvent, out of the 10 selected solvent mixtures under study, with regards to the removal of oily and fatty matters, colouring matter as well as the resinous matter from the crude cane wax. This resulted in producing a light coloured granular hard wax having a high m.p., greater hardness, and light yellowish colour.

In quality testing in an open system, the crude wax and solvent have been required in the ratio of 1:10, and 8-10% has been observed to be the evaporation loss of the solvent. This light yellowish colour hard wax obtained, has been decolourized
using air alone or oxygen gas which resulted into a light cream colour granular hard wax having the properties almost similar to the imported waxes such as carnauba, ouricuri etc.

Bench Scale Study

On the basis of the results obtained on lab. scale, a bench scale pilot plant, fully made of glass, has been designed, and installed in the lab., and having features such that the cold and hot extractions can be carried out in the same unit with arrangements of recovery and recirculation of solvent, resulting into a much more reduced quantity of solvent requirement. The unit has a jacketed reaction column connected at the bottom to the two necked receiving flask/boiler attached with a lagged vapour line tube inclined at the upper end and this end is connected at the junction of two condensers, one upper, as a reflux condenser for escaping out the uncondenseable gases, and the second lower, a jacketed condenser, which is connected to the head of reaction column through the solvent reservoir.

The unit has been designed for treatment of 150 gms. of crude cane wax and manufacture of 100 gms. hard wax per charge. The fractionation of crude cane wax have been experimented on bench scale pilot plant installed as per arrangement shown diagrammatically in Fig. A, using the most suitable selected solvent as azeotropic mixture (B.P. 80.8°C) of isopropyl alcohol (B.P. 82.5°C) and sym. dichloroethane (B.P. 85.7°C) in the ratio of
THE BENCH SCALE PILOT PLANT FOR FRACTIONATION OF CRUDE SUGARCANE WAX.

FIG. A
1:3, as per the flow process shown in Fig. B and the separation is achieved at 60% hard wax and 32-33% soft wax.

**Flow Process Vis-a-Vis Process Developed**

**Separation of Hard and Soft Wax**

*(Defatting and Deashing)*

The crude wax is converted into flakes of 3-4 mm. size by melting the crude wax pebbles obtained by grinding the wax slabs. These wax flakes are packed in the jacketed reaction column. This jacketed column is connected at the bottom to the receiving flask wherein the elute from the column is collected and heated for taking out solvent as vapours. The condenser is provided in the vapour line to condense the vapours in such a way that the condensed vapour falls in the column through a reservoir under control.

In the 1st stage, the azeotropic mixture is added and the temperature of the column and the solvent entering into it, is maintained at 20°C by circulation of water in the jacket of the column. The soft wax dissolves and elutes out into the bottom flask, from where the solvent is evaporated by heating the mass at 82°C. The vapours are condensed through the condenser at 20°C by circulating water in the jacket of condenser and the condensed solvent is recirculated. This system is continued till there is no trace of fatty matter coming into the elute. Then the circulation is stopped and the vapours are allowed to be condensed and stored in the reservoir till the soft wax is free of solvent.
FLOW PROCESS – FRACTIONATION OF THE CRUDE CANE WAX.

FIG. B
The soft wax is then taken out.

In the IIInd stage i.e. after removal of soft wax from the crude cane wax, the whole process is repeated in exactly the same manner as above except that hot water at 85-90°C is circulated in the jacket of column and condenser from the thermostat to maintain the temperature of the mass (Defatted wax) in column and of condenser at 82°C. Since the hard wax dissolves out at about 80-82°C and elutes into the bottom flask wherein the solvent is removed from the dissolved hard wax by evaporation. The hard wax is taken out. Meanwhile the resinous matter remaining in the reaction column, is taken out.

This process has resulted in producing a very light coloured granular and crystalline hard wax having quite firm jel formation property, good oil retention power and high m.p. with a very good fractionation as indicated in the attached table.

Decolourization

Mostly the colouring matter in the cane wax are the colours derived from the cane and these are chlorophylls, carotenoids, anthocyanins and other pigments etc.

The hard wax, obtained by eluting it after removal of the soft wax, is of light yellowish in colour, since most of the colours are removed with the soft wax extraction, wherein the crude wax is treated with sym. dichloroethane in the mixture. This solvent is rich in chloride radicals which are responsible
# TABLE

**Bench Scale Pilot Plant Experiment**

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Properties</th>
<th>Hard wax</th>
<th>Soft wax</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Colour</td>
<td>Light Yellowish</td>
<td>Dark Brown</td>
</tr>
<tr>
<td>2.</td>
<td>Melting Point</td>
<td>79–83°C</td>
<td>51–55°C</td>
</tr>
<tr>
<td>3.</td>
<td>Specific Gravity (25°C)</td>
<td>0.994</td>
<td>-</td>
</tr>
<tr>
<td>4.</td>
<td>Penetration</td>
<td>0.90</td>
<td>-</td>
</tr>
<tr>
<td>5.</td>
<td>Ash</td>
<td>0.0475</td>
<td>-</td>
</tr>
<tr>
<td>6.</td>
<td>Acid Value</td>
<td>9.87</td>
<td>28.91</td>
</tr>
<tr>
<td>7.</td>
<td>Saponification Value</td>
<td>69.39</td>
<td>95.11</td>
</tr>
<tr>
<td>8.</td>
<td>Iodine Number</td>
<td>9.79</td>
<td>34.82</td>
</tr>
<tr>
<td>9.</td>
<td>Ester Value</td>
<td>59.52</td>
<td>66.20</td>
</tr>
<tr>
<td>10.</td>
<td>Acetyl Number</td>
<td>43.31</td>
<td>-</td>
</tr>
<tr>
<td>11.</td>
<td>Unsaponifiable Matter</td>
<td>80.23%</td>
<td>-</td>
</tr>
<tr>
<td>12.</td>
<td>Ratio Value</td>
<td>6.03</td>
<td>-</td>
</tr>
</tbody>
</table>
for bleaching and removal of colour, but still some of the colour is left over which comes in hard wax. The removal of these colours can be done by using potassium dichromate, cromic acid etc., as has been done by earlier workers, the use of which will involve drastic chemical treatment and so has to be avoided and not used in this process. So, herein it has been resorted only to the oxidation by bubbling oxygen or air.

The experiments on oxidation by oxygen as well as by air, indicated a little difference and so oxidation by compressed air has been preferred in order to cut down the cost. The oxidation yielded a very light cream colour while maintaining the granular structure and other properties of hard wax.

**Brief Norms of the Process**

The process developed bears the following norms:

1- The raw crude cane wax has to be flaked 3-4 mm. size.
2- The flakes are to be packed in the jacketed tower.
3- At initial stage, it has to be treated by flowing azeotropic binary solvent mixture, maintaining the ratio (wax : solvent:: 1:2) by recycling the vapours.
4- The azeotropic mixture employed, is of isopropyl alcohol and symmetrical dichloroethane in the ratio of 1:3.
5- In cold system, the soft wax is extracted. Extraction achieved to the tune of 32-33% of the crude wax.
6- After the extraction of soft wax, the temp. of the whole
system is raised to 80°C and the digestion of hard wax is carried out by the same binary mixture while recirculating the recovered solvent till the hard wax is extracted. Extraction obtained to the tune of 60% of crude wax.

7- The pitch, 7%, is removed and discarded.

8- The elute and distillation of solvent vapours are maintained in a sealed state in order to avoid the loss of solvent by evaporation. Although the loss of evaporation is very little, yet a safe margin of 1% loss has been taken into account. The requirement of solvent for treatment of 100 Kg. of crude wax, comes to 1.8 litres.

9- The decolourization requires compressed air (10-15 lb/inch²) to be passed in hard wax in a molten state.

Required Features of Commercial Plant

The required units are GRINDER for making pebbles of crude wax, and then melting it into a MELTER. The molten wax is dropped in cold water bath in a FLAKER. The flakes are dried in a DRYER and then packed in TOWERS for COLUMNAR extraction.

For a continuous operation three such towers are required. These towers are jacketed columns in which arrangements are there, for circulation of cold water during soft wax extraction and of hot water (85°C) during hard wax extraction.

All the three towers are provided with three individual
sets having each two necked jacketed heating stills of which one neck is connected to the bottom of the column and the other to the vapour line. The top of the vapour line is connected at the junction of two condensers, i.e., one to serve as reflux condenser and the other as vapour condenser, which is connected to head of the jacketed column through a reservoir.

These three sets provide an arrangement for continuous operation. The full cycle in each column is of 6 hrs., which comprises filling of the tower - soft wax extraction - hard wax extraction - discharge of pitch, and continuous evaporation of the solvent from the eluates, condensing the vapours through the condensers, collecting it in the reservoir for continuous recycling.

The three columns run, one on filling, other on soft wax and third on hard wax service cycles. This way the process is continuous. The assembly is depicted in the enclosed Fig.C.

**Economic Feasibility**

**Economics For One Ton Of Hard Wax Production Per Day**

**CAPITAL INVESTMENT**

<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Plant and Equipment</td>
<td>Rs.15,01,500.00</td>
</tr>
<tr>
<td>B. Building, Sites, etc.</td>
<td>Rs.20,00,000.00</td>
</tr>
<tr>
<td>C. Recurring Investment</td>
<td>Rs. 4,80,000.00</td>
</tr>
<tr>
<td>D. Working Capital</td>
<td>Rs.10,00,000.00</td>
</tr>
<tr>
<td><strong>Total Investment, say</strong></td>
<td>Rs.50,00,000.00</td>
</tr>
</tbody>
</table>
SCHEMATIC ARRANGEMENT FEATURES OF THE COMMERCIAL CANE WAX REFINING PLANT

FIG. C

Drawn by RPShukla
Approved by Sivashanmugan
<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Raw Materials and Chemicals</td>
<td>Rs. 58,000.00</td>
</tr>
<tr>
<td>B. Utilities</td>
<td>Rs. 3,000.00</td>
</tr>
<tr>
<td>C. Labour and Supervision</td>
<td>Rs. 300.00</td>
</tr>
<tr>
<td>D. Overheads</td>
<td>Rs. 4,000.00</td>
</tr>
<tr>
<td>E. Packaging and Forwarding</td>
<td>Rs. 4,700.00</td>
</tr>
<tr>
<td><strong>Total cost per day</strong></td>
<td>Rs. 70,000.00</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Hard Wax, 1080 Kg.</td>
<td>Rs. 59,400.00</td>
</tr>
<tr>
<td>cost of production @ Rs. 55/- per Kg.</td>
<td></td>
</tr>
<tr>
<td>B. Soft Wax, 576 Kg.</td>
<td>Rs. 10,600.00</td>
</tr>
<tr>
<td>cost of production @ Rs. 18.45 per Kg.</td>
<td></td>
</tr>
<tr>
<td><strong>Total produce</strong></td>
<td>Rs. 70,000.00</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Description</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Hard Wax, 1080 Kg.</td>
<td>Rs. 70,200.00</td>
</tr>
<tr>
<td>selling price @ Rs. 65.00 per Kg.</td>
<td></td>
</tr>
<tr>
<td>B. Soft Wax, 576 Kg.</td>
<td>Rs. 14,400.00</td>
</tr>
<tr>
<td>selling price @ Rs. 25.00 per Kg.</td>
<td></td>
</tr>
<tr>
<td><strong>Total credit/day</strong></td>
<td>Rs. 84,600.00</td>
</tr>
</tbody>
</table>
PROFITS

Profit per day ... Rs.14,600.00
Profit per year (@ 300 days working) ... Rs. 43,80,000.00
Return on Capital ... 86%

Utilities of the Product

The hard wax so obtained will certainly replace the imported —— waxes such as carnauba, ouricuri, candelilla, Japan and Montan waxes, and save the foreign exchange. The following are the industries where cane wax so produced will find its use:

Hard wax

1. Boot, leather, floor, metal polishes.
2. Carbon papers, laminations and tapes.
3. Hardening material for candles
4. Textile and match-box impregnation.
5. Rubber compositions.
6. Grinding polishes, sticks and compounds.
7. Emulsions for fruit preservations and as a barrier for insecticides.
8. Moulding compositions
10. Cosmetics
11. Insulating tapes and compounds for electrical goods and such many other uses.
Soft wax

1. Manufacture of paint.
2. Production of anti-corrosive metal coating compositions.
3. In leather dressings as a finishing and softening agent.
4. For lubrication.
5. Manufacture of industrial greases.
6. Manufacture of polishing and grinding pastes and polishes.
7. Preparation of sex hormones such as progesterone from the sterols particularly, stigmasterol present in good percentage in non-saponifiable fraction of the soft wax.

If cane wax industry is developed in India, not only it will save Rs. 6-7 crores worth of foreign exchange but also can earn at least ten times more than this amount by its export.

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