10.0 Extraction of crude wax from sugarcane press mud:

10.1 Historical Background:

Although sugarcane has been growing as a commercial crop for many years, the whitish powdery deposit did not draw proper attention of chemists till 1840. According to Dr. Balch, a Chemist, Avequin of New Orleans (USA) isolated cane wax and purified it and gave it the name "CEROSIN" from cera meaning wax. The more practical aspects of sugarcane wax recovery seem to have been based upon the investigation of S. Wijnberg who studied the properties of cuticle wax in some details as well as the wax product which he found to be present and recoverable in large amounts in the crude waste press cake (clarification mud) of cane sugar factories. It has been observed that when sugarcane is crushed, the waxy coating of the stem of sugarcane partly becomes dislodged and is dispersed, both mechanically and in true colloidal suspension in juice. During cane juice clarification, the wax suspension is coagulated and the particles adhere to heavy insoluble lime salts and are thus concentrated in the mud. This is very convenient from the point of view of possible manufacture of wax from sugarcane because this concentration is a normal part of the sugar process and hence costs nothing from the point of view of wax processing.
In the year 1916, the first commercial scale plant for extraction of wax from filter cake was established at Durban, Natal (South Africa). But some time in the year 1928, this plant was shut down due to various difficulties encountered and uneconomic prices fetched for the wax.

Walter Scott reported that sugarcane wax was recovered from press mud Java (presently, Indonesia) during approximately the same period as in South Africa. The bulk of the crude cane wax produced in Java was exported to Greece, where it was employed in the manufacture of the brown candles used in Churches. The recovery of crude wax in Java ceased as a result of the devaluation of Greek currency and consequent disappearance of the only market for the crude wax. There were also three other plants producing crude wax from sugarcane mud, one at Nambour in Australia and two others in Cuba. The Australian plant at Nambour lost its economic viability in 1960 (Paturau, 1969).

In Cuba at Central Delicias and then Central Chaparra, commercial production of sugarcane crude wax was started in 1944 and 1947 respectively. As per latest information available, in Cuba there has been a small wax refining plant in operation since 1982 producing refined wax approximately 15-20 tonnes/year. A new plant with a capacity of 500 tonnes/year of refined sugarcane wax was scheduled to begin operation in 1990 as information available in the 'Hand Book of Sugar Cane Derivatives', published by the Group of Latin American and Caribbean Sugar Exporting countries, Mexico.
In India, a plant for the extraction of crude wax from sugarcane mud and refining of the same wax was established at Ravalgaon in the year 1947 employing batch extraction process. Due to some unavoidable circumstances, this plant was stopped after 1980. Another plant was set up in India in the year 1960 by the K.C.P. Ltd. This plant was supplied by Rose Downs & Thompson Ltd., England. Plant capacity was 1.7 tonnes of air dried filter cake per charge. The total capacity was 3.4 tonnes air dried press mud per day. However, as the demand for crude sugarcane wax could not be developed, this plant had to be closed down in 1965 (Kulkarni 36 (1990). This plant has again started commercial production as reported by Manohar Rao 37.

10.2 Factors affecting the wax content in cane:

The quantity of wax which is present in press mud will clearly depend upon many factors among which following are obvious:

- the variety of cane ground;
- the severity of milling process;
- the type of filtration employed, this is whether filter presses or vacuum filters are used;
- the temperature of maceration water;
- the method whereby the trash is removed from the cane i.e., whether or not the cane is subjected to burning;
- the climatic condition under which cane is grown;
- the method and efficiency of clarification and mud subsidation.
All the above factors will have an effect on wax yield and it is important to remember that all are the possibility of variations.

10.3 Reasons for closure of wax plants in different countries:

The main reasons for the closure of these plants in different countries are:

- very low recovery of hard refined wax compared to Carnauba wax;
- recovery of solvent was very low;
- quality of crude wax and refined wax produced from press mud was not up to the mark of industrial use;
- removal of fat from wax in order to get a hard wax like carnauba wax was proving very expensive;
- Carnauba wax was not having the above problems and hence manufacturers preferred the use of Carnauba wax.

From the above, it is evident that the crude wax extracted from sugarcane press mud needs some modification in its characteristics for having industrial quality like that of carnauba wax.

11.0 Background of crude wax extraction from press mud:

Extraction of cane wax from filter cake has been reported by many workers. Normally, press mud obtained in sugar factories is used in the field by farmers as manure. Direct application of press mud to soil as manure as is being practiced now, the wax present might deteriorate
the physical properties such as permeability, aeration and soil structure and composition etc., and with the passage of time this deterioration might get worsen. Hence removal/degradation of wax from filter cake before direct application in soil would be helpful to enhance the quality of filter cake as organic manure.

11.1 Solvents used in crude wax extraction from press mud:

The recovery of crude wax from press mud is a very simple process. Cane wax being an organic matter easily soluble in organic solvents. Naturally the most scientific methods so far developed for extracting cane wax from filter mud is by adopting solvent extraction process. The only constituents of press mud soluble in non-hydroxylic solvents such as benzene or petroleum hydrocarbons are the waxes, fatty materials and pigments. Hence it is only necessary to dry the filter cake and perform a solvent extraction in order to recover the crude wax. The most important point is the selection of best solvent, and the least expensive solvent, due to the reason that in any solvent extraction process, there is bound to be some loss of solvent. A number of solvents have been found suitable for extraction of crude wax from press mud. These are ethyl ether, acetone, benzene, ethyl alcohol, carbon tetrachloride, chloroform, petroleum ether, methyl alcohol, a mixture of ethyl ether and alcohol, petroleum naptha etc. The molecular formula, specific gravity as also their boiling points are as under:

<table>
<thead>
<tr>
<th>Common name</th>
<th>Molecular formula</th>
<th>Specific gravity</th>
<th>Boiling point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table-XXIV</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Substance</td>
<td>Molecular Formula</td>
<td>Density</td>
<td>Viscosity</td>
</tr>
<tr>
<td>----------------------------</td>
<td>-------------------</td>
<td>---------</td>
<td>-----------</td>
</tr>
<tr>
<td>Acetone</td>
<td>C₂H₃COCH₃</td>
<td>0.792</td>
<td>56.5</td>
</tr>
<tr>
<td>Benzene</td>
<td>C₆H₆</td>
<td>0.879</td>
<td>80.1</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>CCl₄</td>
<td>1.595</td>
<td>76.8</td>
</tr>
<tr>
<td>Chloroform</td>
<td>CHCl₃</td>
<td>1.651</td>
<td>112.3</td>
</tr>
<tr>
<td>Ethyl alcohol</td>
<td>C₂H₅OH</td>
<td>0.789</td>
<td>78.4</td>
</tr>
<tr>
<td>Diethyl ether</td>
<td>C₂H₅-O-C₂H₅</td>
<td>0.708</td>
<td>34.6</td>
</tr>
<tr>
<td>Methyl alcohol</td>
<td>CH₃OH</td>
<td>0.792</td>
<td>64.7</td>
</tr>
<tr>
<td>Mineral turpentine</td>
<td>C₁₀H₁₆</td>
<td>0.868</td>
<td>150-190</td>
</tr>
<tr>
<td>Hexane</td>
<td>C₆H₁₄</td>
<td>0.659</td>
<td>66-70</td>
</tr>
<tr>
<td>Petroleum ether</td>
<td>C₅H₁₂-C₇H₁₆</td>
<td>0.640</td>
<td>35-70</td>
</tr>
<tr>
<td>Toluene</td>
<td>C₆H₅CH₃</td>
<td>0.870</td>
<td>108.4-112.4</td>
</tr>
</tbody>
</table>

It has been observed by various authority including the author that the aromatic hydrocarbons have higher extraction efficiency and they extract more wax than the aliphatic hydrocarbons, in fact benzene itself was found to be the most efficient solvent. Petroleum hydrocarbons may, however, be selective in their action on filter mud. However, experiments conducted revealed that shell solvents in a required boiling range preferred as these are available from refineries directly at a much cheaper rate. Shell solvent and solvent oil in the boiling range of 90-120 °C are preferable, because the solvent oil in this boiling range is mostly heptane which is a very good solvent for cane wax. For extraction of crude wax from press mud much work has been done and patents have been patented for economical recovery of wax. However, very less work has been done in regard to crude wax hardening to make it suitable for industrial use.

11.2 Extraction Process:

Normally the press mud obtained from vacuum filter containing about 70% moisture is dried to about 10% moisture in order to reduce the loss of extractable matter through fermentation and to avoid the obnoxious smell of the end product due to the presence of moisture. Though the drying of vacuum filter mud can be effected by different methods and equipment, the most economical method is sun drying, which is adopted normally in the sugar factories. At Ravalgaon Sugar Factory, sun-drying method was followed and mineral turpentine was found to be the most suitable solvent.
11.3 Temperature of extraction:

Crude wax is soluble in the solvents in all proportions, particularly at temperatures above the melting point of wax, which is within the range of 50 °C to 80 °C, depending on its composition. On the basis of the trials conducted in laboratories and plants, a temperature of 70 °C to 80 °C was found to be most appropriate.

11.4 Extraction equipment:

The most common equipment used by the plants that worked in South Africa, Australia, Cuba and India is the batch type of solvent extraction equipment. The flow of the solvent is usually downwards by gravity as the filter cake is charged on to a perforated screen plate covered with gunny bags at the bottom of the conical vessel so that the cush cushion / fine unwanted particles are not mixed with the solvent and percolated through. The gunny bags are normally soaked with sufficient quantity of water prior to the start of the process of extraction of wax so that it cannot absorb/retain any solvent. The vessel is then filled up with solvent and then heating with steam starts several times till no wax is left behind in the press mud. Subsequently, the wax is recovered from the solvent by evaporation and the vapour thus evaporated is condensed back to solvent for reuse in the process again.

Normally the ratio of solvent to filter cake is about 3:1. The continuous solvent extraction plants now available for solvent extraction of vegetable oils would be the ideal equipment for this purpose, but they are
expensive and unless a plant is bound to work continuously for some years, this type of plant may prove highly expensive. This again depends on the market for cane wax on a long term basis, at reasonable prices.

11.6 A flow diagram of crude wax extraction unit is given as under:

FLOW DIAGRAM

Dry press mud

| Ground to coarse powder

| Solvent extraction

(n-hexane, acetone, toluene or turpentine)

| Extract

| Distil

| Residue (Exhausted cake)

| Steam

| Solvent (Reused)

| Crude wax Vapour (Condensed, water phase removed and and solvent regenerated and reused)

| Exhausted cake (Used as bulk manure)
12.0 Characteristics and composition of sugarcane wax:

Wax obtained from press mud by the solvent extraction method of dry press mud/wet press mud as mentioned above is known as crude sugarcane wax. The general characteristics and composition of sugarcane crude wax as indicated by different authorities is as under:

According to Vidyarthi and Narasinga Rao: 39

Table-XXV

- Melting point: 66 – 78 oC
- Saponification value: 60 – 135
- Acid value: 15 – 23
- Iodine value: 26-31.5
- Acetyl value: 61 – 90

Saponifiables:

- Free and combined acids: 53.0%
- Caproic acid: 0.3 %
- Palmitic acid: 14.7%
- Stearic acid: 11.8%
- Oleic acid: 22.0%
- Arachidic acid: 1.8%
- Resinous matter: 2.4%
- Unsaponifiables: 43.7%
Crude sugarcane wax when fractionated by means of ethanol-benzene mixture separates into hard wax and fatty matter. According to Abdal-Akher et al., 1977, the characteristics and composition of crude sugarcane wax are as under:

**Table-XXVI**

- Melting point 66-78°C
- Saponification value 60 – 135
- Acid value 15-23
- Iodine value 26-31.5
- Acetyl value 61-90

**Saponifiables**
- Free and combined acids 53.0%
- Caproic acid 0.3%
- Palmitic acid 14.7%
- Stearic acid 11.8%
- Oleic acid 22.0%
- Arachidic acid 1.8%
- Resinous matter 2.4%
- Unsaponifiables 43.7%
Crude wax obtained from dry press mud (by Sulphitation process) by solvent extraction method by using Turpentine as solvent, on analysis gave the following characteristics and composition (S.C.Ray 41', 1986):

**Table-XXVII**

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour</td>
<td>Dark green</td>
</tr>
<tr>
<td>Smell</td>
<td>Disagreeable</td>
</tr>
<tr>
<td>Melting point (°C)</td>
<td>75 – 77</td>
</tr>
<tr>
<td>Acid value</td>
<td>9.15</td>
</tr>
<tr>
<td>Saponification value</td>
<td>200 – 250</td>
</tr>
<tr>
<td>Iodine value</td>
<td>32 – 40</td>
</tr>
<tr>
<td>Ester value</td>
<td>191 – 235</td>
</tr>
<tr>
<td>Specific gravity</td>
<td>0.97 - 0.98</td>
</tr>
<tr>
<td>Ash content</td>
<td>2 – 3%</td>
</tr>
<tr>
<td>Unsaponifiables</td>
<td>20 – 50</td>
</tr>
</tbody>
</table>

Rao and Gupta et al. 42, (1941) observed the following constants for the crude wax:

**Table-XXVIII-**

<table>
<thead>
<tr>
<th>Petroleum solvent (used)</th>
<th>Other solvent (used)</th>
</tr>
</thead>
<tbody>
<tr>
<td>--------------------------</td>
<td>-----------------------</td>
</tr>
<tr>
<td>Solvent 50</td>
<td>80 100 110</td>
</tr>
<tr>
<td>used to</td>
<td>to to to</td>
</tr>
<tr>
<td>60</td>
<td>90 110 120</td>
</tr>
</tbody>
</table>

From the above experimental results, it is observed that the physical and chemical constants of the wax extracted from the press mud vary considerably with the type of solvent used. This is not surprising when it is considered that the wax is not a single component, but a mixture of several other organic compounds like glycerides of different fatty acids, alcohols of esters. Further, the composition and the quality of wax would vary with the type of cane, soil etc. Same observations were made by the author at the time of extraction of crude wax in the laboratory from press mud at Ravalgaon.
Nigam and Raha et al. 43 (1973) observed that during extraction of wax press mud must not be too finely pulverized, since this would require a long settling time after extraction. On the other hand if the particles are very coarse, percolation is too rapid requiring an excessive quantity of solvent and extending the time to obtain a reasonable recovery. The solvent should percolate at a temperature as near as its boiling point as possible, a few degree above the melting point of wax. The boiling point of solvent should not be too high to create the difficulty of its recovery.