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**WAX AND FAT FROM SUGARCANE PRESS-MUD**

MUHAMMAD QUDRAT-I-KHUDA AND NAIMUDDIN AHMED,

*East Regional Laboratories, Pakistan Council of Scientific and Industrial Research, Dacca*
**Introduction.**

Different workers<sup>1</sup> have isolated wax from sugar-cane press-mud. This wax appears as a white deposit on each node of the sugar-cane and passes into the mud which accumulates during the preliminary purification of cane juice and contains almost the entire quantity of fatty and waxy substances amounting to 15% of the total weight of the mud.

In East Pakistan from one mill alone about 2000 tons of this mud is thrown away every year which could yield 300 tons of crude wax.

The magnitude of this waste could be realised from the fact that some 6 sugar factories have already started work in East Pakistan and 6 more are expected to come into being in the next few years.

No work seems to have been done on the recovery of the wax from sugar-cane press-mud of East Pakistan factories. There is, therefore, a necessity of finding of ways and means to separate the wax and make pure product out of it. This has now been done and the results are being described in this communication.

The method of recovery of the wax that has now been adopted differs considerably from those of earlier workers. The recovery of crude wax was effected with petroleum. The hot extract on cooling, yielded a solid, which we call sugarcane wax. Some more of it could be obtained, when petroleum is completely removed and the residue treated with rectified spirit. The solution in spirit, on removal of the solvent, yielded the fatty constituent. This fat, which has been called sugarcane fat was found to contain glycerides of oleic, palmitic and stearic acids. On hydrolysis of the sugarcane wax, melissic acid ( $C_{30}H_{60}O_2$ ) and myricyl alcohol ( $C_{30}H_{62}O$ ) were obtained. From the fatty constituent, on hydrolysis, a small amount of non-saponifiable matter was also isolated which appears to be steroid in character, and melts at 139°C. Further studies of this substance are still in progress.

Some of the physical and chemical constants of the wax have been given in the Table below, which shows clearly the differences that the present sample of the wax and fat bear compared to the samples prepared by others in the past<sup>2</sup>.

**COMPARISON OF THE CHARACTERISTICS OF SUGARCANE WAX AND FAT**

	Wijnberg	Lewko- witsch	Bosz	Vidyarthi and Rao	Present sample	
					Wax	Fat
Saponification value	167.9	81.2	177.0	133.5	100.2	140
Iodine value	60.0	87.0	..	31.5	1.0	44
Acid value	38.6	11.9	47.3	23.4	0.0	12.8
Nonsaponifiable matter..	58.8	69.1	..	43.7	58.4	8.8
Melting point	55.79°	58.59°	60—62°	68.7°	85°	Semi-solid.

### Experimental

The dried and powdered press-mud (1000 g.) was extracted with hot petroleum ether (b.p. 80-100°C.) in a soxhlet apparatus, when the crude wax came out in petroleum as a green solution. From the petroleum extract of the crude wax, on cooling, 65 gms. of a white solid product crystallised out. From the mother liquor, the solvent was distilled off, and the residue, on crystallisation with rectified spirit, gave another 37 gms. of the same solid. This brought the total quantity of solid to 102 gms., constituting 10.2 percent of the weight of dry press-mud. This solid product was repeatedly crystallised from petroleum ether (b. p. 80-100°C.) when a wax melting at 85°C. was obtained. This has been called sugar-cane wax, and constitutes 68 percent of the total crude wax mixture. The mother liquor was treated with animal charcoal, when it lost the green colour and was obtained as a light brown liquid, which, on cooling became a semi-solid mass. This weighed 47 grams and consisted of a mixture of partly solid fat and a liquid oil. This mixed product has been described as sugar-cane fat, and constitutes 4.7 per cent of the weight of the dry mud, which brings the quantity of the crude extract to 14.9 per cent.

An alternative procedure for the purification of the wax was also tried with better results. The petroleum extract of the crude wax was completely freed from the solvent, and the residue dissolved in the minimum quantity of hot benzene. To this was added rectified spirit, which, on cooling, precipitated out the white sugarcane wax, and the product was further purified by repeating the same process i.e. by dissolving the mass in benzene and reprecipitating the solid wax by rectified spirit. This method is definitely less cumbersome and can thus save time in purification considerably.

#### Sugarcane wax

Sugarcane wax (50 grams) was dissolved in benzene (200 c.c.) and saponified by refluxing with an excess of 5 N alcoholic caustic potash (200 c.c.) for three hours, and then the benzene-alcohol mixture was removed as completely as possible over a water bath and under water

pump. The residual soap was taken in water and refluxed several times with petroleum ether and benzene successively to remove unsaponified product completely. The clear soap solution was then acidified and the liberated acid was taken up in warm petroleum ether. Ultimately, after the removal of solvents, 19.6 gms. of a fatty acid and 29.2 gms. of the resulting alcoholic product were obtained.

On repeated crystallisation from benzene-alcohol mixture, the acid fraction melted at 84-85°C., and gave a neutralisation value of 121.7 and an iodine value of 1.2.

The methyl ester of the acid was prepared in the usual way, which melted at 70 to 71°C. 0.2 grams of the silver salt of the acid on heating gave 0.0388 grams of silver; the molecular weight of the acid is, therefore, 449.7. M. W. required for  $C_{30}H_{60}O_2$  is 452. These definitely suggest the acid to be melissic acid.

The alkali insoluble product (29.2 gms) was repeatedly crystallised from a mixture of benzene and alcohol when it melted at 86-87°C. This was analysed for myricyl alcohol. Found: C, 82.0 per cent; H, 14.0 per cent.  $C_{30}H_{22}O$  requires C, 82.19 per cent and H, 14.16 per cent. This was treated with acetic anhydride in presence of anhydrous sodium acetate, when the acetate of the alcohol was formed, which crystallised from benzene-alcohol mixture and melted at 75-76°C. Some quantity of the alcohol was oxidised with chromic acid, when a product melting at 84-85°C. was obtained; the latter was found to be melissic acid.

The sugarcane-wax can, therefore, be said to be myricyl melissate. On analysis, it gave C, 82.2 per cent; H, 13.5 per cent, where  $C_{60}H_{120}O_2$  requires C, 82.54 per cent and H, 13.76 per cent.

#### Sugarcane Fat

The sugarcane fat (25 gms.), obtained from the mother liquor of sugarcane wax, was hydrolysed with caustic potash (17 grams) in alcoholic solution, when it gave 22.2 grams of a fatty acid mixture and 2.2 grams of non-saponifiable matter in the usual way. The glycerin produced in the reaction was not isolated.

The acid fraction (22.2 grams), which was a mixture of saturated and unsaturated acids, gave a neutralisation value of 203.0 and an iodine value of 56.4. The separation of the acids was effected by Twitchell's Method through the lead salt as modified by Hilditch.<sup>3</sup> The process was repeated until complete separation was obtained. The unsaturated acid fraction (13.6 grams) that was thus separated gave a neutralisation value of 199.0 and an iodine value of 90.0. To find out its exact composition a further separation through urea complex was undertaken. The whole of the acid was dissolved in methanol which was already saturated with urea. The urea complex of the acids<sup>4</sup> crystallised out in only one crop. The yield was almost quantitative, showing thereby the presence of only one kind of unsaturated acid. A little of the acid was subjected to oxidation with potassium permanganate by Lapworth Mottram Process<sup>5</sup> when dihydroxy stearic acid, melting at 129-30°C., was obtained. Thus, it is to be concluded that the only unsaturated fatty acid in this sugarcane fat is oleic acid.

The saturated acid fraction (8.6 grams) gave a neutralisation value of 215.4, and an iodine value 1.6. It melted at 50-51°C. 0.3405 grams of silver salt of the acid gave on decomposition 0.10 grams of silver; the molecular weight of the acid is, therefore, 260.7. This suggests that the solid acid is probably a mixture of about 82.5 per cent of palmitic acid and 17.5 per cent of stearic acid. This is also supported by its neutralisation value.

The non-saponifiable matter (2.2 grams or 8.8 per cent) was crystallised several times from benzene-alcohol mixture, when it came out as shining crystals melting at 139°C. These were found to respond to the Leibermann-Burchard Test and were assumed to be sterols. Further examination of this substance is now in progress.

#### Discussion

The separation of the fat and wax can be more completely effected, as stated, with benzene and alcohol mixture. The solvent from the mixture is regenerated with ease, and thus, the process promises to be compar-

tively inexpensive for large scale operation. The fatty acids, that can be made out of the sugarcane fat fraction, can be put to more than one use.

From the experiments described above, it is apparent that for every 100 tons of the press-mud, there will be ten tons of sugarcane-wax and about five tons of sugarcane fat available for processing into polishes and soap. It is expected that a total quantity of some 2,000 tons of wax and 1,000 tons of fat may thus be put to use immediately and a like amount to be available when more of the sugar factories begin to work. This is not a fact to be ignored, particularly when we are not very well supplied with wax and fat.

#### Summary

The examination of the sugarcane press-mud of East Pakistan sugar factories shows that out of its total quantity it can yield some 15 per cent of a wax and fat mixture, called sugarcane wax and sugarcane fat, of which wax is about two-thirds and the rest is fat. The wax is the ester, myricyl melissate, while the fat is a mixture of the glycerides of oleic, palmitic and stearic acids.

#### Acknowledgement

Our thanks are due to Dr. S. Siddiqui, Director, Pakistan Council of Scientific and Industrial Research, for his lively interest and encouragement during the progress of the work.

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