

OBSERVATIONS ON THE FERTILIZER VALUE AND WAX CONTENT OF FILTER CAKE.

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The necessity for having more information about filter cakes in this country has been felt for a long time. The reasons for this investigation become more apparent with the introduction of Oliver Campbell filters, the acute fertilizer shortage in the Union, and the possibilities of putting the cane wax contained in the filter cake to some industrial use, as was done in the last war.

Until recent years only two main types of filter cakes were found in the industry, namely, carbonation cake from the Natal Estates factory, and sulphitation cake from all other factories. As is well known, the carbonation filter cake is higher in lime, but lower in phosphates and other ingredients than the sulphitation cakes, which themselves vary considerably in chemical composition. The Oliver filter has now introduced further variations in the composition of cakes from the sulphitation factories, owing, of course, to the dilution with bagacillo. Approximately half of the factories in Natal and Zululand have now installed the Oliver filters, and very little information is available on the comparative qualities of cakes from the old and new processes.

Regarding the fertilizer value of filter cakes, the present time appears to be opportune for an investigation of this nature. With the shortage of artificial fertilizers it is of special importance to use additional sources of plant-food. Karroo manure, for example, has recently received much attention in the Union, but filter cake has the advantage of being available within the industry itself.

Filter cake has been extensively used for agricultural purposes, and has proved itself by field experiments to be a very useful fertilizer medium on most types of soil. For this reason alone it was felt that a more comprehensive survey of the fertilizer ingredients was very desirable.

A further benefit resulting from the work contemplated in this paper is that comparative filter cake analyses from the factories are placed on record for the first time. Not only should such analyses be of value to the chemical control of each factory, but should be of interest to chemists and agriculturists alike.

All the samples analysed below were collected from the factories over the month of August, 1941, and were intended to be representative of that month.

The following table gives the available constituents, soluble in 2 per cent. citric acid, as is usual for fertilizers. For convenience total nitrogens are given in the same table.

TABLE I.
Available Constituents and Total Nitrogens (N) of Filter Cakes from Natal and Zululand Sugar Factories.
(Results on air-dry basis.)

Factory.	Soluble in 2 per cent. citric acid solution.				
	Total nitrogen (N) per cent.	Available Phosphoric Oxide (P_2O_5) per cent.	Available Potash (K_2O) per cent.	Available Lime (CaO) per cent.	Available Magnesium Oxide (MgO) per cent.
1. The Illovo Sugar Estates, Ltd.	0.80	2.96	0.24	15.98	0.88
2. Prospecton Sugar Estates, Ltd.	0.91	2.24	0.40	9.15	1.22
3. Reynolds Bros. Ltd., Sezela...	0.70	2.04	0.13	14.23	0.50
4. New Guelderland Sugar Factory	0.70	2.80	0.15	11.50	0.62
5. Zululand Sugar Millers and Planters, Ltd.	0.79	3.16	0.16	11.85	0.78
6. Sir J. L. Hulett & Sons, Ltd., Darnall	0.63	2.50	0.12	12.43	0.67
7. The Tongaat Sugar Co., Ltd.	0.84	2.10	0.12	12.73	1.27
8. Sir J. L. Hulett & Sons, Ltd., Amatikulu	0.77	2.50	0.13	17.63	0.86
9. The Natal Estates, Ltd.	0.30	0.71	0.11	40.65	0.97
10. Melville Sugar Co., Ltd.	0.87	2.74	0.14	15.87	1.00
11. Gledhow-Chakas Kraal Sugar Co., Ltd., Chakas Kraal	0.63	1.30	0.29	20.18	0.77
12. The Umfolozi Co-op. Sugar Planters, Ltd.	0.85	3.20	0.25	17.25	1.00
13. The Doornkop Sugar Estates, Ltd.	0.81	1.80	0.17	9.37	0.51

Factory.	Total nitrogen (N) per cent.	Soluble in 2 per cent. citric acid solution.			
		Available Phosphoric Oxide (P_2O_5) per cent.	Available Potash (K_2O) per cent.	Available Lime (CaO) per cent.	Available Magnesium Oxide (MgO) per cent.
14. Glendale Sugar Co.	1.05	2.98	0.49	8.77	0.46
15. Umzimkulu Sugar Co., Ltd.	1.17	3.00	0.25	14.46	0.81
16. Central Factory, Ltd.	0.81	2.58	0.09	10.85	0.54
17. Sir J. L. Hulett & Sons, Ltd., Felixton	1.01	3.00	0.11	18.02	1.04
18. Gledhow-Chakas Kraal Sugar Co., Ltd., Gledhow	0.71	2.60	0.16	13.70	1.15
19. The Entumeni Sugar Milling Co. (Pty.), Ltd.	0.64	2.64	0.16	11.13	0.48
20. Reynolds Bros., Ltd., Esperanza	0.82	1.68	0.09	15.00	0.58
21. Crookes Bros., Ltd., Renishaw	0.90	2.12	0.12	13.10	0.57
Average (excepting Natal Estates)	0.81	2.50	0.19	13.69	0.70

From the above table it can be seen that the total nitrogen ranges in the sulphitation factories from 1.17 per cent. to 0.63 per cent., with an average of 0.81 per cent. This figure compares very favourably with the total nitrogen content of Karroo manure, which ranges from about 1.4 to 2.0 per cent. nitrogen. It compares still more favourably with the total nitrogen in composts, which ranges from roughly 0.4 to 1.2 per cent.

Available phosphoric oxide (P_2O_5) is, as may be expected, very high in the filter cakes, the Zululand factories averaging the highest with almost 3 per cent. P_2O_5 .

Potash (K_2O) is, of course, present in negligible amounts, a dressing of 10 tons filter cake per acre supplying only about 40 lbs. K_2O . The potash analyses, however, were made by way of interest, as questions regarding the potash content of filter cake may occasionally be asked.

Available lime (CaO) and available magnesia (MgO) were estimated also largely by way of interest, as these are not generally regarded as plant-foods. That calcium and magnesium are absorbed in considerable quantity by sugarcane has been amply demonstrated from time to time, and it may be that these elements do play a more important part in plant nutrition than we expect. While the lime content is far in excess of the plant's requirements, it is useful as a soil rectifier, increasing the pH of very acid fields and improving the soil structure of other fields.

It was thought that a more detailed knowledge of the composition of filter cake would be unconstructive, and in consequence four factories were selected at random for a complete analysis. Had time been available it was intended to analyse all the filter cakes in greater detail. The following four results, however, will serve as a guide to those interested:—

TABLE II.
Total Analysis of Filter Cake, Expressed as Per Cent. Oven-dry Substance.

Ingredient.	Reynolds Bros. Ltd. Esperanza.		Crookes Bros. Ltd. Renishaw.		Entumeni Sugar Milling Co. (Pty.) Ltd.		Gledhow-Chakas Kraal Sugar Co. Ltd., Gledhow.	
	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.	per cent.
Silica (SiO_2)	3.67	5.53	2.97	5.01	2.97	5.01	2.97	5.01
Lime (CaO)	20.57	16.28	18.03	15.14	18.03	15.14	18.03	15.14
Magnesia (MgO)	0.75	0.74	0.68	1.47	0.68	1.47	0.68	1.47
Potash (K_2O)	0.10	0.15	0.18	0.17	0.18	0.17	0.18	0.17
Phosphoric Oxide (P_2O_5)	2.24	2.53	3.55	3.22	3.55	3.22	3.55	3.22
Titania (TiO_2)	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Oxide of Manganese (MnO)	0.02	0.03	0.03	0.02	0.03	0.02	0.03	0.02
Oxide of Iron (Fe_2O_3)	0.45	0.59	0.47	0.50	0.47	0.50	0.47	0.50
Alumina (Al_2O_3)	2.22	2.59	4.41	3.65	4.41	3.65	4.41	3.65
Soda (Na_2O)	0.07	0.10	0.09	0.09	0.09	0.09	0.09	0.09
Sulphate (SO_3)	20.66	15.86	19.30	11.49	19.30	11.49	19.30	11.49
Loss on ignition	48.65	55.28	50.19	59.01	50.19	59.01	50.19	59.01

These results show that for the four factories a little more than 80 per cent. of the phosphoric oxide, lime and magnesia is soluble in 2 per cent. citric acid, and almost all the potash. As the period of shaking is only 30 minutes, this indicates a high degree of solubility for the plant-foods.

The above analyses indicate that we have here in the industry a by-product which should be of great value as a fertilizer. Assuming that some 80,000 tons of dry filter cake are produced each season, then we have at our resources about 700 tons nitrogen (N), equivalent in fertilizer value to about 3,000 tons ammonium sulphate, 2,000 tons phosphoric oxide (P_2O_5), or 12,000 tons superphosphate, and 150 tons potash (K_2O) or 300 tons muriate. These large quantities of valuable fertilizer material, if carefully and judiciously applied to the fields, should go a long way towards solving our fertilizer problems, particularly during this period of national emergency.

There is in filter cake a relatively high proportion of sugarcane wax which regularly goes to waste. In the present time of need, attention is once more given to this substance, with the result that a number of experiments have been made here to ascertain the percentage composition of crude sugarcane wax in filter cake from the various factories, and the most economic and suitable method of extraction. The sugarcane wax obtained in Natal during the last war was reported to be "sticky, dark in colour, difficult to bleach and often malodorous." Recently,¹ in 1940, a survey was made of the wax content of Louisiana filter cakes, with very encouraging results. The sugarcane wax obtained there melted at 174°F. (the melting point of carnauba wax being 180°F.), and in many other characteristics it resembled carnauba wax, which in its unpurified condition is obtained from the leaves, berries and stalks of a palm growing in Brazil and other parts of South America.

The pioneer and largely successful efforts made in Natal during the last war to extract sugarcane wax from filter cake appear to have attracted much attention. Thus Kingzett's Chemical Encyclopaedia defines sugarcane wax as "a by-product extracted in Natal and Java." "Topics and Comments" in the "Manufacturing Chemist" for December last also refers to sugarcane wax as "a South African innovation of the last war" and "hopes that every attempt will be made to 'repatriate' the idea."

The war has turned attention to this wax and for three seasons cane wax has been studied in Louisiana and reports have been issued.² The American Navy, finding the usual sources of supply closed, have caused investigations to be undertaken into methods of extraction and refining cane wax, and a pilot plant has been set up for this purpose. Cane wax can also find a market in South Africa for military purposes.

The chief obstacle to making the by-product a commercial success is that the filter cakes from many of the factories have now been diluted greatly with bagacillo by the use of the Oliver filter, as mentioned earlier in this paper. The filter cake is consequently more bulky and difficult to handle. Despite this difficulty, it is hoped that the possibility of obtaining the wax commercially will not be abandoned. As a preliminary towards the study, a number of analyses were made in the laboratory at the Experiment Station, using benzene as the solvent, and extracting in a Soxhlet for five hours. The results are appended below.

TABLE III.

Sugarcane Wax Content (Benzene Extract) of Filter Cakes.

(Results expressed as per cent. oven-dry filter cake.)

1. Illovo Sugar Estates, Ltd.	4.90
2. Prospecton Sugar Estates, Ltd.	9.36
3. Reynolds Bros., Ltd., Sezela... ..	6.82
4. New Guelderland Sugar Factory	3.72
5. Zululand Sugar Millers & Planters, Ltd.	2.79
6. Sir J. L. Hulett & Sons, Ltd., Darnall... ..	4.09
7. The Tongaat Sugar Co., Ltd.... ..	3.41
8. Sir J. L. Hulett & Sons, Ltd., Amatikulu	5.21
9. The Natal Estates, Ltd.... ..	0.59
10. Melville Sugar Co., Ltd.	4.48
11. Gledhow-Chakas Kraal Sugar Co., Ltd., Gledhow	4.91

12. The Umfolozi Co-operative Sugar Planters, Ltd.	2.62
13. The Doornkop Sugar Co., Ltd.	5.30
14. Glendale Sugar Co., Ltd.	12.80
15. Umzimkulu Sugar Co., Ltd.	9.06
16. Central Factory, Ltd.	2.38
17. Sir J. L. Hulett & Sons, Ltd., Felixton	4.56
18. Gledhow-Chakas Kraal Sugar Co., Ltd., Chakas Kraal	7.47
19. The Entumeni Sugar Milling Co. (Pty.), Ltd.	0.65
20. Reynolds Bros., Ltd., Esperanza	3.46
21. Crookes Bros., Ltd., Renishaw	4.96

The average sugarcane wax content from the above table is 4.93 per cent., ranging from a maximum of 12.80 per cent. to a minimum of 0.59 per cent. Apart from about five factories, however, the wax content of the filter cake does not vary considerably from the mean. It appears that factories with the Oliver filter have lower wax content than the other factories. Umfolozi and Sezela are exceptions to this rule. It is interesting that many factories with complete chemical control give lower benzene extracts:

Considerable work has been done in the past on the wax content of sugarcane. Of the various solvents used from time to time (chloroform, acetone, carbon bisulphide, ether, carbon tetrachloride, paraffin, benzene, alcohol, to name a few), some would not be commercially available. Before continuing this study it is therefore essential to select the most suitable solvent. Alcohol was tried and yielded considerably higher results than benzene. Examination of the extract, however, revealed the presence of much sugar and perhaps some salts, all of which, of course, would have to be removed.

SUMMARY.

The need for a systematic study of the fertilizer and wax content of filter cake is stressed.

Analyses of available plant-foods of composite filter cake samples from all the factories are given and discussed. A total analysis of four samples is given for comparison, and attention is drawn to the large amount of plant-food available to the industry.

The content of wax in sugarcane is discussed and analyses (benzene extract) are given in support.

The need for further work on the subject is stressed.

Reference.

¹ Industrial Reference Service (1941), 76. Quoted in "Manufacturing Chemist" (1941), 12, 263.

² Fort, C. A. (1938): "Determination of Sugarcane Wax in Louisiana," The Sugar Bulletin 17, No. 4, 4; see also The Sugar Bulletin (1939), 17, No. 20, 3; see also The Sugar Jour. (1941), 4, No. 6, 24.

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Mr. DU TOIT said that he was indebted to the reporter of a local newspaper for the following information: A considerable source of supply of wax was now closed to the United States. At the request of the U.S. Navy a search for new sources was undertaken. The result was that chemists found a way of extracting sugarcane wax cheaply and a pilot plant was set up to determine the cost under actual manufacturing conditions. Several million pounds could be obtained annually from sugarcane processed in the United States.

Acotinic acid, too, was essential in the manufacture of many of the plastics vitally needed for war industries. The present process of producing it from citric acid was costly. It constituted the principal organic acid in sugarcane. The best source to recover it from was molasses. Investigation showed that relatively large quantities occurred in cane tops. The acid occurred in combination with other substances, and American Government chemists were now working on ways of separating it commercially. Here again it is expected that the available supply would run into millions of pounds.

Mr. DYMOND said that when scums were boiled up to ease filtration, a portion of the cane wax became emulsified and passed into the boiling house. The process used by the Natal Cane By-Products at the end of the last war consisted of a benzene extraction from dried Illovo filter cake. The first wax produced started melting at 50°C. and became totally liquid at about 72°C. Buyers who used it for floor and boot polishes, required a harder wax, so it was fractionated with ether to remove the fats. The principal trouble, however, was its colour, and no satisfactory method was ever devised for bleaching it. Cane wax in its natural state was white to pale yellow, but the effect of the sugar clarification process caused a black coloration. He suggested that the only way to obtain this wax in a white or light yellow condition would be to extract it from Peck strainer cush-cush, or the scums from the old-fashioned wax separators which operated prior to clarification. Such a procedure would, however, be difficult owing to the high sucrose content of such products.

Mr. Dymond recommended that the wax investigation should be continued.

Mr. BIJOUX said that he had carried out certain experiments and found that a white wax could be extracted from filter cake with a certain solvent. He could not disclose the solvent, but felt sure that the interests concerned would give the Experiment Station all the help in this connection if approached.

Mr. Bijoux said that the low percentage of wax in filter cakes coming from factories using the Oliver Campbell type of filter was probably due to the liberal use of hot water, which dissolved precipitated wax formed, and returned it into process.

Mr. RAULT said that he had carried out some wax determinations some years ago, using different solvents. He found the wax content of filter cake much higher. Most factories ranged from 7 to 12 per cent., while Natal Estates had 2 per cent. wax. Factories that were trying to cut chemicals showed a higher percentage wax in filter cake, but the actual elimination was much the same. Mr. Rault thought that an appreciable quantity of wax was carried away in the cush-cush where screen filters were used. This might account to a certain extent for the low wax content now obtained.

He was rather surprised that not more magnesium was found in Natal Estates filter cake. Not only was more lime used, but being an alkaline process, the magnesium elimination was almost perfect.

The VICE-PRESIDENT said that some years ago a seedling was grown at the Experiment Station that was exceptionally rich in wax. There was a thick coating of wax on the outside of the sticks which made them resemble candlesticks. The seedling was discarded, however, as it was thought that it would not be acceptable to the mills.

Mr. MOBERLY thought that the use of cold imbibition water might account to a certain extent for the lower wax content of filter cake.

Mr. DYMOND agreed with Mr. Moberly and said that the Oliver Campbell filters might also be partly responsible for the lowering of the wax content of filter cake, as they allowed more wax to become emulsified and passed into the boiling house, where it gave trouble. He would have preferred a low temperature filtration. He did not think the new varieties had much, if any, effect on these figures.

Mr. Dymond said that the wax investigation should be continued and those interested should assist the Experiment Station. It would be appreciated if Dr. Tromp, Director of War-time Research, or any Government officials, could collaborate.

Mr. DODDS said that he had a letter from India in which it was stated that juice was treated with a suitable solvent before clarification so as to extract the wax. This process ensured the maximum elimination of wax and improved clarification.

Mr. VIGER pointed out that low temperature filtration, especially with the Oliver Campbell filters, would be disastrous. Deterioration would take place rapidly and the purity of the filtrate would drop. A considerable amount of hydrogen sulphide would be formed and this would ruin copper screens in four months, and even stainless steel screens would tarnish and corrode rapidly. He had found by analysing both mixed and clarified juice that 99 per cent. wax was eliminated at the filter station. Low temperature filtration was therefore dangerous and unnecessary.

Dr. HEDLEY, in reply, said that he did not think that there would be much profit in wax extraction, but wax was an article that was urgently needed under present conditions, and the whole problem should be tackled at once.