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RECOVERY OF CARBONATES AND HYDROXIDES FROM COCOA POD ASH : ANALYSIS OF SAMPLES FROM OCHISO LIQUID SOAP PILOT PLANT

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Abstract

Ashes of cocoa pods contain alkali which is traditionally leached out and used to produce 'alata' soap. Ghana's Institute of Industrial Research has tested a pilot plant that produces liquid soap from cocoa pod ash, waste lime ($Ca(OH)_2$), and palm kernel oil by initially converting the potassium carbonate in the leachate to hydroxide. Percentage recovery of K_2CO_3 in the leachate averaged 50.2 per cent, while 75.9 per cent of $Ca(OH)_2$ was recovered with a conversion rate of 86.3 per cent. Pilot plant conversion figures compared favourably with earlier laboratory work (85.0 - 85.7 %), but the results suggest efforts be made to improve the filtration system of the pilot plant.

Résumé

MENSAH, B. & AGGEY, M.: *Récupération de carbonates et de hydroxydes de cendre de la cosse de cacao: Analyse d'échantillons de l'installation pilote du savon liquide à Ochiso.* Les cendres de cosses de cacao contiennent alcali qui est traditionnellement lessivées et utilisées pour produire le savon 'alata'. L'Institut Ghanéen de Recherche Industrielle a soumis à l'essai une installation pilote produisant le savon liquide de cendre de la cosse de cacao, la chaume usée ($Ca(OH)_2$) et l'huile d'amande de palme par la conversion initiale du carbonate de potassium dans le filtrat lessiviel à hydroxyde. Le pourcentage de récupération de K_2CO_3 dans le filtrat lessiviel avait la moyenne de 50.2 pour cent alors que 75.9 pour cent de $Ca(OH)_2$ était récupéré avec une proportion de conversion de 86.3 pour cent. Les chiffres de conversion d'installation pilote comparaient favorablement avec les résultats du travail précédent de laboratoire (85.0-85.7 %) mais les résultats suggèrent que les efforts doivent être fait pour améliorer le système de filtration de l'installation pilote.

Introduction

Plants often selectively remove minerals, especially potassium, from the soil, thereby acting as potash concentrators. Thus, ashes of various agricultural wastes, including cocoa pods and shells, palm bunches and plantain peels, contain appreciable amounts of alkali as carbonates. Traditionally, the ashes have been used as sources of alkali for the production of '*alata*' soap. To diversify the product spectrum of such agro-wastes and introduce a new product line for the small scale traditional soap manufacturers, a technology was developed at the CSIR-Institute of Industrial Research to produce liquid soap from the ashes of these agricultural wastes. A pilot plant was accordingly set up at Ochiso, a village in the Central Region of Ghana. The unit operations and processes involved leaching, causticization, concentration, saponification, and soap finishing.

This paper reports on the efficiency and effectiveness of the leaching operation and the causticization process.

Experimental

Principle

Leaching ashes of woody matter with water results in an alkali solution consisting mainly of

potassium carbonates. Causticization of this leachate with $Ca(OH)_2$ is represented by the equation:

$$K_2CO_3 + Ca(OH)_2 \rightleftarrows 2KOH + CaCO_3$$

Potassium hydroxide is filtered off, as $Ca(OH)_2$ and $CaCO_3$ are insoluble. The yield recovery and the extent of conversion of K_2CO_3 to KOH are then evaluated. The potassium hydroxide solution is then used to saponify palm kernel oil to produce soap. Fig. 1 shows the process flow diagram.

Materials

Cocoa pod ash was purchased from the Cocoa Research Institute at Tafo in Ghana. Lime was acquired as industrial waste from L'air Liquide Ltd, an acetylene-manufacturing company in Tema, Ghana.

Preparation of samples

Using ratios of ash, water and lime determined from previous work in the laboratory, a specified quantity of water was measured into a leaching

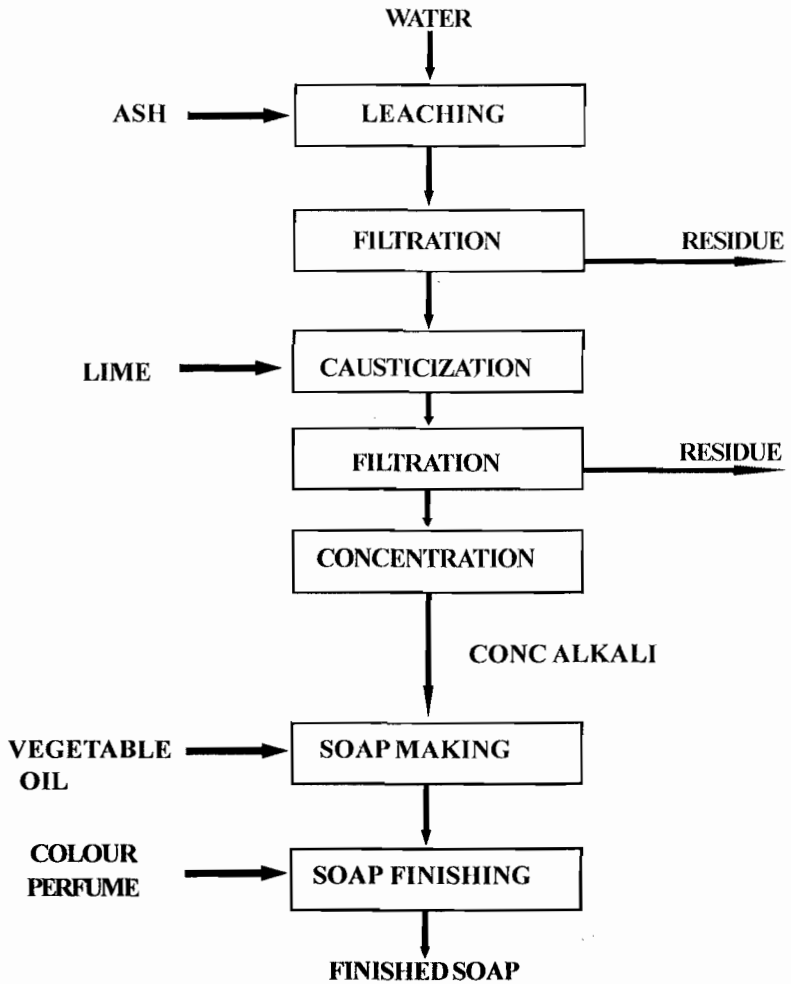

Fig. 1. Process flow diagram for liquid soap production.

vessel and the stirrer switched on. A specified quantity of ash was also weighed and added to the water in the vessel. The mixture was stirred continuously for 1 h and then filtered under gravity. The volume and alkali content of the filtrate were determined. The leached liquor was heated to 95-100 °C, and a specified quantity of waste lime was added to the hot, leached liquor with continuous stirring at 95-105 °C for 2 h. It was then filtered after cooling to below 50 °C. The volume of filtrate as well as its carbonate and hydroxide contents were then determined.

In the preceding laboratory work, leaching and causticization were done simultaneously in the same reaction vessel. However, to process the lime residue further for additional income, the two operations were separated in the pilot plant.

The caustic liquor was then concentrated to 10-12 per cent hydroxide before it was used to saponify the palm kernel oil to produce liquid soap. The test runs were repeated to confirm the results.

Determination of alkali content of ash

About 4.00 g of ash was weighed and its moisture content determined by drying at 105 °C to constant weight. About 2.00 g of the dry ash was weighed and reacted with a specified quantity of 0.23 M HCl, and the excess acid titrated against 0.03 M NaOH. The amount of acid used to neutralize the alkali in the ash was determined, and the percentage of alkali in ash was then calculated as potassium carbonate.

Determination of alkali content in leachate

Ten millilitres of the leached liquor was pipetted into a 100-ml volumetric flask and made up to the mark with distilled water. Twenty millilitres of this solution was titrated against 0.1 M HCl, using methyl orange indicator. The alkali content was calculated as potassium carbonate.

Analysis of caustic liquor

Ten millilitres of the caustic liquor was pipetted into a 100-ml volumetric flask and made up to the mark with distilled water. Ten millilitres of this solution was titrated against standard 0.1 M HCl, using methyl orange as indicator. The volume of acid used corresponds to the hydroxide and carbonate contents of the solution.

Another 20 ml of the solution was pipetted into a 100-ml volumetric flask and diluted with 20 ml of distilled water. The carbonate in the solution was precipitated with 10 per cent barium chloride solution. After complete precipitation, the solution was made up to 100 ml with distilled water and then filtered. Fifty millilitres portion was then titrated against standard 0.1 M HCl, using phenolphthalein indicator. The titre corresponds to the hydroxide content of the caustic liquor.

Results and discussion

Analysis of ash

The ashes of various flora have been found to contain appreciable amounts of alkali mainly in the form of potassium with minor quantities of sodium. Neelam Jalil *et al.* (1991-1996) reported that the ashes of palm tree trunk contain 319.4 mg g^{-1} of potassium (or 56.6 % K_2CO_3) and 13.4 mg g^{-1} of sodium (or 3.09 % Na_2CO_3). Acquah (1999) reported that potassium is the major cation in cocoa bean shell ash, containing 126.2 mg g^{-1} potassium; the paper did not report on the sodium content. Furthermore, the ash was reported to contain 138.2 mg g^{-1} carbonates, 4.43 mg g^{-1} nitrates, 1.54 mg g^{-1} phosphates, and minor quantities of chlorides and sulphates. In this study, the total alkali in the ash was calculated as K_2CO_3.

Leaching process

Table 1 presents the results of the leaching process. The carbonate content in the leached liquor ranged from a maximum of 0.069 kg l^{-1} to a minimum of 0.053 kg l^{-1}, with an average of 0.059 kg l^{-1}.

The percentage recovery of K_2CO_3 in the leachate, based on the K_2CO_3 content of the ash, also varied from a maximum of 55.2 per cent to a minimum of 47.5 per cent. The differences in the

TABLE 1

Recovery of carbonates from leached liquor

Batch no.	*Input*		*Output characteristics*			
	Ash (kg)	*Water (l)*	*Filtrate (l)*	*[K_2CO_3] (kg/l)*	*Carbonate recovered (kg)*	*% carbonate recovered*
1	30	120	96.0	0.069	6.624	55.2
2	30	120	107.5	0.053	5.698	47.5
3	30	120	107.0	0.055	5.885	49.0
5	30	120	105.0	0.056	5.880	49.0
6	30	120	101.0	0.060	6.060	50.5
Average	30	120	103.3	0.059	6.029	50.2

yield are partly due to the ineffectiveness of the filtration method used, as seen in the variation in the volumes of filtrate (Column 4 of Table 1). Notwithstanding this, the average yield of 50.2 per cent is quite low. Potassium carbonate is very soluble in cold water, with a standard solubility of 105.5 g/100 g water (Lang, 1961). It is, therefore, expected that all the alkali in the ash will be leached out into solution.

The low yield is probably due mainly to the physical nature of the bulk of the ash as received from an ashing kiln. The ash contains particles of varied sizes, at times with very hard chunks of particles which do not easily leach into water. Grinding the ash to uniformly smaller particle size could increase the recovery. However, the cost of grinding will have to be factored into the total cost of production of the final liquid soap.

Causticization

Table 2 shows the results of the causticization process. Equation 1 represents the chemical reaction of potassium carbonate in the leachate with lime ($Ca(OH)_2$):

$$K_2CO_3 + Ca(OH)_2 \rightleftarrows 2KOH + CaCO_3 \qquad (1)$$

The hydroxide concentration of the caustic liquor, calculated as potassium hydroxide, ranges between 0.042 and 0.050 kg l^{-1}. The unreacted carbonate content in the liquor also ranges between 0.0070 and 0.0090 kg l^{-1} (Table 2). The average concentrations are 0.045 and 0.0090 kg l^{-1}, respectively.

The extent of conversion of carbonates to hydroxide ranges from 86.2 to 88.3 per cent (Table 2). This compares favourably with a conversion of 85.0 per cent, using water to ash ratio of

TABLE 2

Conversion and recovery of hydroxide after causticization

Batch no.	*Input*	*Output characteristics*				
	Leached liquor (l)	*Filtrate (l)*	*[KOH] (kg/l)*	*[K_2CO_3] (kg/l)*	*Conversion* % %*	*% hydroxide recovery**
1	96.0	62.0	0.043	.0070	88.3	66.4
2	107.5	63.0	0.044	.0083	86.8	69.0
3	107.0	81.0	0.042	.0082	86.0	82.5
4	105.0	75.5	0.050	.0090	87.3	90.6
5	101.0	77.5	0.045	0.0081	87.2	78.4
Average	103.0	71.8	0.045	0.0081	87.2	78.4

3.75:1.00, and 85.7 per cent with water to ash ratio of 5:1 recorded in a previous laboratory study (Mensah, 1988).

The amount of hydroxide recovered from causticization ranges between a yield of 63.6 and 90.6 per cent. The differences in the amount of hydroxide recovered, as reflected in the amount of filtrate recovered from the various batches, are attributable to the inefficiency of the filtration system used and the retention of caustic liquor in the lime sludge. The recovery of hydroxide in the laboratory was 96 per cent.

Conclusion

The extent of conversion of carbonate to hydroxide in the pilot plant compares well with that of laboratory work, though filtration under laboratory conditions was better controlled, resulting in yields higher than that in the pilot plant. The results suggest that effort should be made to improve the filtration system to increase recovery.

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