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A Novel Process for The Production of Potash from Plant Ash: Leaching Technique

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ABSTRACT

The leaching process involved in the production of Potash from plant ash was investigated. Three samples viz palm inflorescence, sunflower stalks and corn stalks were selected, as the raw ash material for the leaching. Analysis of the solution resulting from the ash shows that sunflower stalks, palm inflorescence, corn stalks contain by weight 43.01 g and 28.19 g Potassium oxide (K_2O) and a total solute content of 65.87 g, 63.61 g respectively. All other material that are not soluble in water are considered inert. Laboratory batch leaching experiments on the ash show that, the optimum leaching temperature for all the samples is 70 ^{0}C for a particle size range of 295 nm to 45 nm. Extraction data on over-flow and under-flow at the optimum leaching temperature and size range gave the number of equilibrium stages to be three for a solvent to feed ratio 3.765, 3.44 and 2.81 for sunflower stalks, palm inflorescence and corn stalks ash respectively. A small scale model of a continuous counter-current leaching system was designed, constructed and tested. An overall stage efficiency of 22.4 % was obtained with the stated solvent to feed ratio. The efficiency of the system was however increased to 83 % by increasing the solvent to feed ratio to 12 times the former.

Keywords: *Counter-current-flow, Leaching, Over-flow, Plant-ash, Under-flow*

INTRODUCTION

Agro-waste or plant biomass in Nigeria are mostly exposed to open-air burning with its associated environmental implications, but these wastes could be resourcefully managed if used for other viable industrial, agricultural, or domestic purposes, thereby decreasing the problem of environmental pollution and also creating wealth [1].

Plant biomass as a source of energy offers a cheap, environmentally friendly alternative to the conventional petroleum energy sources and these energy generated from biomass such as tree bark, wood residues, and other plant materials, produces a considerable amount of fly and bottom ashes. Managing waste ashes generated from plant is a major challenge, as land filling and open dumping are the main options in management of the waste ash.

Cleared wood needed to be disposed of, and the easiest way to accomplish this was to burn any wood not needed for fuel or construction. Ashes from hardwood trees could be used to make lye, which could be used to make soap or boiled down to produce valuable potash. Exploration of ash-derived alkalis for domestic use is an ancient craft [2, 3] involving simple technology and chemistry [4]. The general principle in potash production from ashes involved leaching the ashes with water; the 'leachate' is then evaporated. The residue crude is the crude potash. The quality of the residue depends on the materials used, as well as procedure and equipment as reported by Babayemi *et al*. [4]. Materials for ashes in potash production such as wood [5, 6], plantain and banana peels [2], [7, 8], cocoa pod husk [9], palm bunch [10], and livestock dung [11] have low potash yield. Furthermore, some of those which give high potash yield are not available throughout the year.

Plants contain alkali metals such as potassium, calcium, sodium and magnesium. These metals are present in form of various salts. When the plant matter is subjected to heat, burnt in presence of presence of air, the metals are oxidized to metal oxides. Carbon dioxide produced during burning of carbonaceous matter, combines with these alkali metallic oxide to produce alkali carbonate. Other products formed during burning, in presence of water vapour, are alkali bicarbonate potassium hydroxide as listed in Kumar [12]. These reactions can be expressed as:

$$X_2O\ (s) + CO_2\ (g) \rightarrow X_2C0_3\ (s) \quad (1)$$

$$X_2C0_3\ (s) + H_2O\ (g) \rightarrow CO_2\ (g) + 2XHCO_3\ (s) \quad (2)$$

$$X_2C0_3\ (s) + H_2O\ (g) \rightarrow XOH\ (s) + XHCO_3\ (s) \quad (3)$$

Where X denotes alkali metal

The chemical reaction equilibrium constants for the above three reactions at 298 K are 2.00 × 10^{60}, 2.07 and 0.33, respectively [9]. It is evident by looking at the order of magnitudes of chemical reaction equilibrium constants that reaction (1) predominates in the above reaction scheme.

Olufemi *et al*. [13], refer to leaching as the extraction of a soluble constituent from a solid by means of different solvents: acids, bases, water and chelating agents. However, since acidic medium are advantageous for the dissolution of metals, acids are commonly used for the leaching of heavy metals. Sulphuric acid is the most common leaching agent because of its chemical properties and also its relatively low cost and the rate of leaching depends on the parameters used such as temperature, time, pH, particle size, concentration of lixiviate, slurry density and agitation speed [14, 15].

The process may be employed either for the production of concentrated solution of a valuable solid mineral, or order to free an insoluble solid, such as pigment, from a soluble material with which it is contaminated. The method used for the extraction will be determined by the proportion of soluble constituents present, its distribution through the solid, the nature of the solid and the particle size.

Economically viable ways of using waste ash rather than having to dispose it off have to be investigated. There is a vast body of information on utilization of fly ash (FA) in building/construction, production of aggregates and more recently, for agriculture [16].

This study therefore, seeks to investigate potential use for waste ash residue of plant (sun flower, stalks and corn stalks) all available in Nigeria were used as a source of potash. Factors affecting the leaching of potash (Potassium Salts) from these materials were studied, and a graphical materials balance method was further used to study the leaching of potash from the ashes of these materials.

EXPERIMENTAL PROCEDURE

Materials used are thermometer, laboratory centrifugal pump, retort stand, speed regulator, tanks (drums), muffle furnace, beaker, hot plate, filter paper, volumetric flask, water bath, funnel, and electric oven. The experiment was carried out in the separation process laboratory of the chemical/petroleum engineering department, Niger Delta University, Bayelsa State, Nigeria.

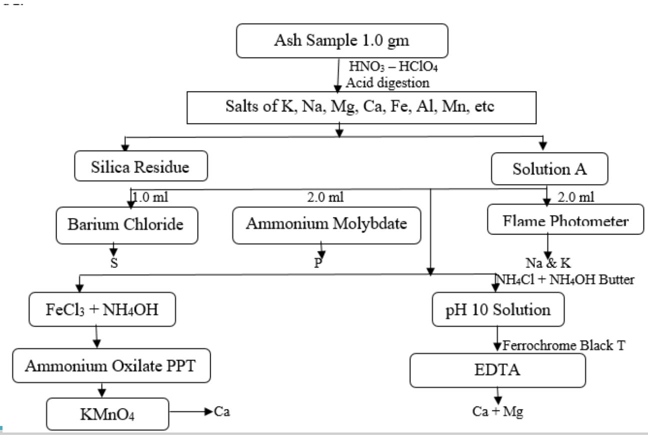
Preparation of the Ash Solution

The preparation of the ash solution for elemental analysis was carried out by Nitric-per chloric acid digestion.

1. 50.0 gm of sun dried sample of plant tissue was weighed and ashed at 44 ^{0}C for 4 hours in a muffle furnace.
2. 1.0 gm of the oven dried ash sample was weighed into a beaker and 20 ml of concentrated HNO_3 acid was added. The beaker was covered with a watch glass and the content digested over hot plate at 180 ^{0}C for 40 minutes to oxidize the organic matter. 10 ml of 60 % per chloric acid was then added and the mixture was heated for 15 minutes to dehydrate the silica. The solution was cooled and diluted to 25 ml with warm water.

The solution was then filtered through a watchman No. 41 paper into a 100 ml volumetric flask. The residue of silica was then washed with 0.5M HCl acid and the solution of diluted to 100 ml volume with distilled water. This solution is labelled A and it contains all the elements to be subsequently determined.

A blank solution was similarly prepared without the sample present. The schematic diagram of these are shown in figures 1 and 2.



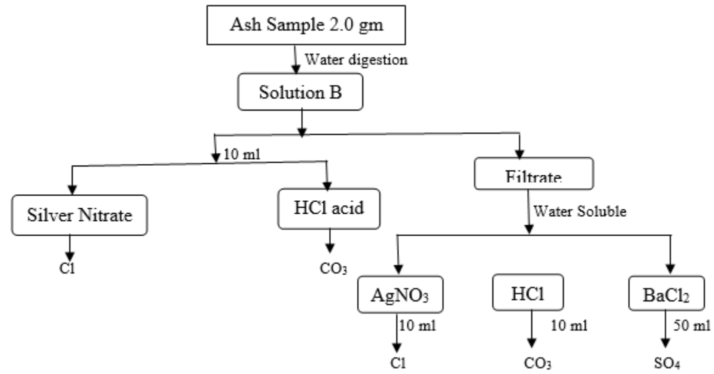


Figure 1. Schematic diagram of procedure 1

Figure 2. Schematic diagram of procedure 2

Locus-of-underflow Concentration

The determination of the relationship between solute concentration in the over flow and under flow and the locus of the under flow concentrations were determined by the method of George [17].

Slurries containing 0.2 to 8.0 gm of the sample in 20 ml of distilled water was prepared and digested for 3 hours over water bath maintained of 70 ^{0}C. The slurry was then separated into under-flow and over-flow by centrifugation and their weight note. The over-flow and under-flow were then analyzed for the solute and solvent content. The compositions of both the over-flow were calculated and then the data obtained from the under-flow concentrations was then used to determine the locus of under-flow concentrations. Using these data the number of stages required in a counter current extraction was determined graphically.

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Pilot Plant Experiments

A counter-current leaching system was arranged as shown in figure 3. The flooding flow rate of the column was determined. A reasonable percentage of the flooding rate, based on experience, was chosen as the operating flow rate of the system using a varied feed to solvent ratio and a temperature of 70 ^{0}C, the water was pumped into the system at the stated flow rate.

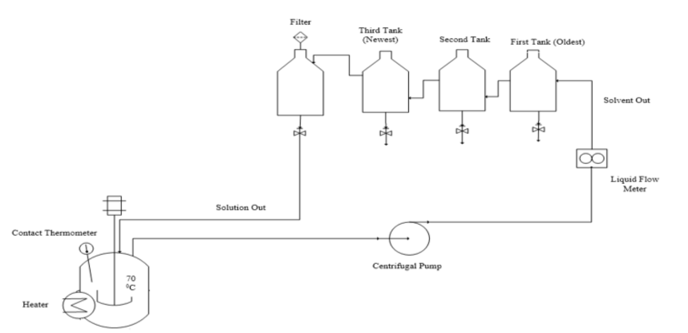

Figure 3. Continuous Counter-current Leaching System

Samples of over-flow solution were withdrawn at intervals and carbonated, chloride and sulphate content of the solution determined. After 4 hours of extraction, the potassium oxide content (K_2O) of the solid remaining in the tanks were determined from which an overall stage efficiency of the system was calculated.

Table 1. Elemental Analysis of the Ash Samples

Ash Constituents	Percentage Weight of Ash Constituents		
	Sun Flower Stalks	Palm Inflorescence	Corn Stalks
Moisture	5.49	3.98	8.93
K_2O	43.01	35.60	28.19
Na_2O	0.63	2.00	0.53
CaO	5.22	7.66	12.62
MgO	4.10	5.24	3.59
P_2O_5	8.17	3.53	3.79
Cl	6.76	4.88	20.30
S	0.94	5.86	0.45
CO_2	24.20	13.20	11.83

RESULTS AND DISCUSSION

Effect of Temperature on Leaching

The analytical methods that were employed in the above methods of analysis were drawn mainly from published literatures pertaining to soil and plant tissue analysis [18–20].

The ashing of the plant tissue was carried out at 440 ^{0}C to prevent the loss of volatile material such as K, Cl, S and P. In the methods of analysis, two different digestion methods were used, the nitric-perchloric acid and water digestion. The use of perchloric acid and other acids in the former precludes the determination of chloride and carbonate from that solution, hence water digestion was employed.

The results of the ash analysis of the three samples are shown in Figure 4. The analysis have shown that, the potash contgent of the three materials is quite appreciable, with sun flower stalks ash having 65.9 wt. %, palm inflorescence 63.6 wt. % and corn stalks 41.0 wt. % potash in the ash.

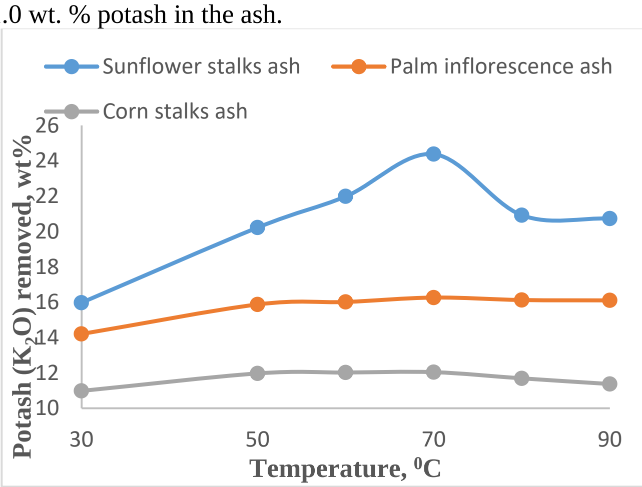

Figure 4: Effect of Temperature on Leaching for batch process

Effect of Particle Size Distribution of Ash Samples

The results of the particle size analysis of the three samples presented in Figure 5. From the result, it is shown that the particle size distribution show that, sun flower stalks ash had the lowest coarse particles (more than 68.0 wt. % of its constituent particles are finer than 75 mm (200 mesh)).

Palm inflorescence ash has more than 43.4 wt. % of its constituent particle finer than 75 mm while corn stalks ash had more than 63 % of its constituent particles coarser than 75 mm.

Effect of Change in Leaching Time

Analysis of the solids (in the tank) after 3 hours of operation showed that, the potash has potassium oxide remaining in the solids are 33.13 wt. %, 27.7 wt. % and 22.23 wt. % for sun flower stalks, palm inflorescence and corn stalk ash respectively. From these values the average overall efficiency is however very low, and by increasing the solvent to feed ratio 12-fold, compared with the optimum ratio, the efficiency of the system was increased to about 83.0 %.

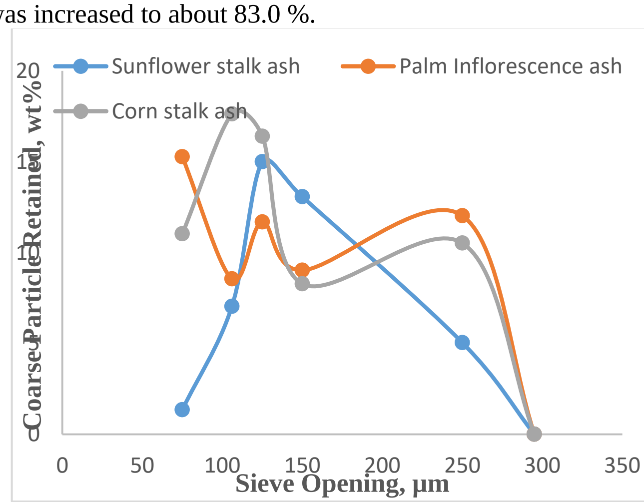

Figure 5: Particle size distribution of ash samples

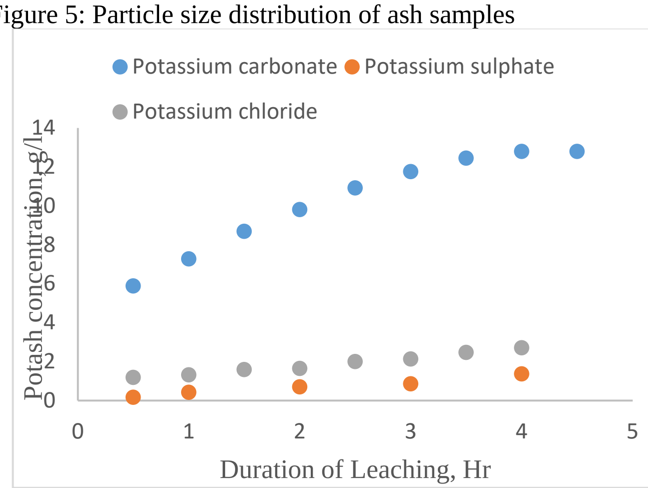

Figure 6: Change in potash concentration with time of leaching for sunflower stalk ash

The results obtained under this condition are shown in Figures 6, 7 and 8 and the variation of potash constituent during the process are indicated.

Relationship between Underflow and Overflow Solutes

Results obtained on the analysis of the overflow and underflow of the three samples shows that unequal concentration in the overflow and underflow solutions may have been caused by insufficient contact time and/or preferential adsorption of the solution on the inert solids.

Using Figure 9, this effect is more pronounce in palm inflorescence and sun flower as than from corn stalk ash.

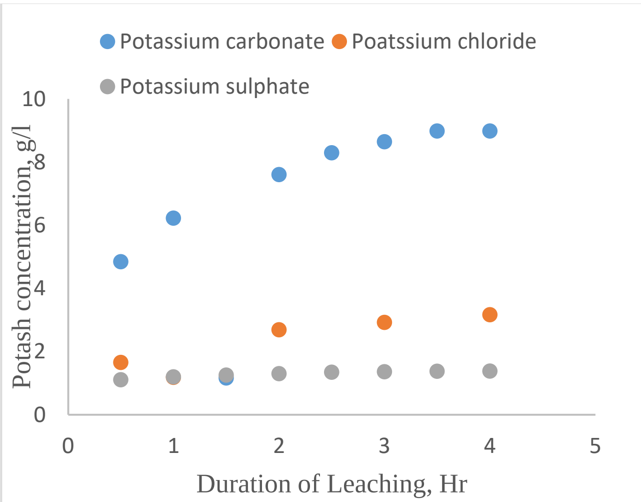

Figure 7: Change in potash concentration with time of leaching for palm inflorescence ash

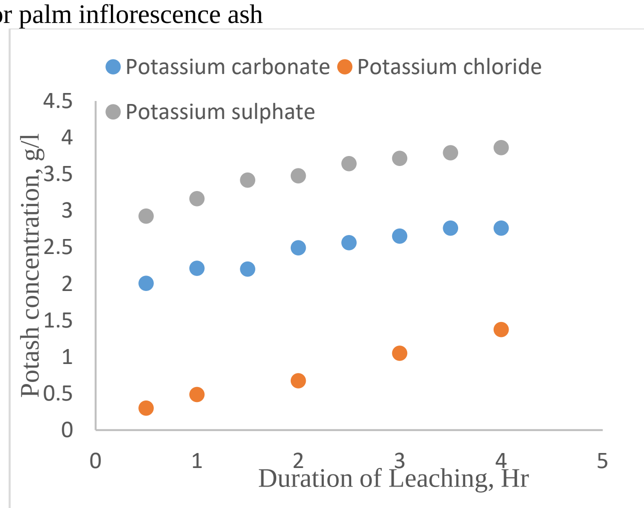

Figure 8: Change in potash concentration with time of leaching for corn stalks ash

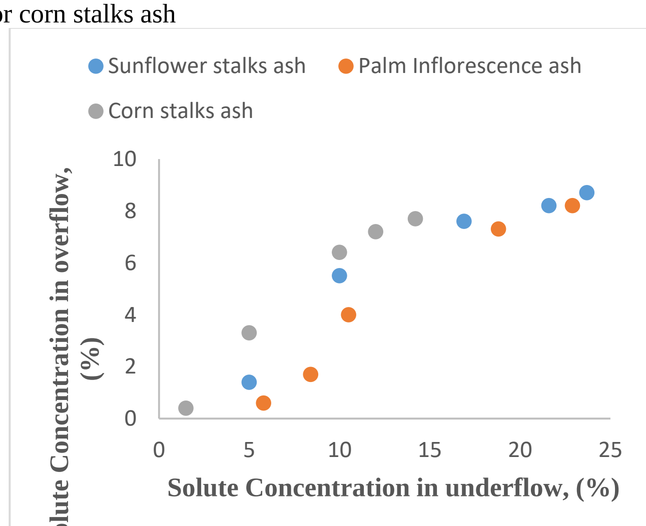

Figure 9: Relationship between solute in the overflow and underflow

CONCLUSION

It can be concluded from the plots above that, the product solution of sunflower stalks and palm inflorescence contain more potassium carbonate (K_2CO_3) than the other constituents, while corn stalks ash contain more potassium chloride in the product. The plots further indicate that, the concentration of K_2CO_3 in the solution approaches saturation exponentially and it remain constant after 3.5 hours of operation for palm inflorescence and corn stalk ash. These results are very useful for scale-up in the informal sectorial operation.

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