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EXTRACTION OF CAUSTIC POTASH FROM COFFEE HUSK: PROCESS OPTIMIZATION THROUGH RESPONSE SURFACE METHODOLOGY

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ABSTRACT

The work investigates ashing of coffee husk and leaching of potassium carbonate from ash as a potential as a source for potassium. The husk was ashed and analysed for ions. It was found that the ash contains a significant amount of alkali metals. Ashing temperature influenced the yield of potassium carbonate, the most predominant alkali in ash. Potassium carbonate was leached from ash by water as a solvent. Variables affecting the yield are the ashing temperature, ash particles size, stirring, and solvent temperature. A 2^3 Factorial design with centre point for experiment design and Response surface methodology was used to study the effect of ashing temperature, ash particle size, and the leaching temperature, which were identified as the key variables. Ash had 71.5 wt. % K when analyzed for alkali metals, and 15.2 wt. % potassium carbonate. Maximum ash yield of 18 wt% was at ashing temperature of 400-500°C. For leaching of carbonate, optimum ashing temperature was 600°C, smallest particles size (40-200 μm), and high solvent temperature (70°C).

Key words: Coffee husk, Leaching, Potassium carbonate, Optimization, Response surface.

INTRODUCTION

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 potassium oxide to produce potassium carbonate. Other products formed during burning, in presence of water vapour, are potassium bicarbonate potassium hydroxide. These reactions can be expressed as:

$$K_2O\,(s) + CO_2\,(g) \longrightarrow K_2CO_3\,(s) \qquad \ldots(1)$$

$$K_2CO_3\,(s) + H_2O\,(g) \longrightarrow CO_2\,(g) + 2\,KHCO_3\,(s) \qquad \ldots(2)$$

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$$K_2CO_3\,(s) + H_2O\,(g) \longrightarrow KOH\,(s) + KHCO_3\,(s) \quad \ldots(3)$$

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

Coffee processing waste

Coffee is processed either by the 'wet' or 'dry' method depending on whether the unwanted matter from coffee berry is removed by using water, or through dry processing.

In the wet process, water is used as an agent to remove coffee pulp consisting of *epicarp* and *exocarp* of coffee berry. The pulp has a very high moisture content (76-88%, on wet basis). Milling of dry parchment coffee produces parchment husk accompanied by *spermoderm* or silverskin. In the dry processing of coffee berry, coffee husks are produced, comprising of pulp and the *endocarp*. Up to 45% (by mass) of coffee berry are made up of husks[2]. Yield of clean *Canephora* (Robusta) coffee is about 22%, based on fresh ripe fruits. One hundred kilogram of fresh ripe fruit yields the following products at various stages of wet and dry processing to produce *Canephora* (Robusta) coffee[3].

(a) Wet Processing

Pulped coffee	74 Kg
Washed coffee	52 Kg
Dried coffee	49 Kg
Pre-dried coffee	44 Kg
Parchment	26 Kg
Clean coffee	22 Kg

(b) Dry Processing

Dry cherries	40-45 Kg
Clean coffee	22 Kg
Fresh pulp	56-60 Kg
Dry pulp, parchment and husk	35-40 Kg

The waste in wet processing are coffee processing effluent and parchment husks. Dry processing produces husk consisting of pulp and *endocarp*. Coffee husks are presently burnt in charcoals burners and industrial boilers to provide thermal energy. It is also used as mulch in farms. Coffee husks are the fourth major agricultural waste in Kenya with a utilization, as fuel, of only 20% (Table 1). About 27000 Tonnes of husks goes unutilized annually, thereby causing problems of environmentally safe disposal[4].

Table 1: Production and use of coffee husks and other non-wood biomass in Kenya

Biomass product	Annual production (Tonnes)	Industrial use (Tonnes)
Maize cob and stalks	1 176 000	-
Bagasse	1 000 000-130 000 000	1 064 000
Saw dust	63 000	7 800
Coffee husks	35 000	7 800
Coconut shell & coir	20 000	-
Cashew nut hulls	9 000	1 300
Rice husks	4 800	-
Sisal waste	4 800	-
Municipal waste	100 000	-

Ash produced from husk is rich in potassium, consisting of 23 wt. % to 44 wt. % of K_2O (or 19 wt. % - 36 wt. % K)[3,5]. If a suitable process is devised to extract this potash, it would have a major impact on economics of coffee production and ease problems of waste disposal.

EXPERIMENTAL

The present study is about ashing of coffee husks and its subsequent leaching by water. Parameters studied are: (i) Effect of ashing temperature on yield of ash, (ii) Effect of stirring speed on yield, (iii) Effect of ashing temperature on fraction of ash leached, (iv) Effect of solvent temperature on yield, (v) Effect of ash particle size on yield. The objective is to identify optimal operating conditions and rate determining step for mass transfer operation of leaching of potassium carbonate.

Coffee husks sample was collected from a coffee processing unit at Ruiru, Kenya. Husk was ashed in a muffle furnace. Water was used as the solvent for extraction. Standard laboratory techniques were used for analytical work. Special equipment/instruments used are indicated under results and discussion.

RESULTS AND DISCUSSION

Elemental analysis of coffee husk ash

Ash was analyzed by Atomic Absorption Spectrophotometer (Chem. Tech. AAS Model CTA2000) for elements potassium, sodium, calcium, magnesium and iron. The average concentration (ppm) is found to be: potassium 41.93, sodium 5.48, calcium 13.75, magnesium 9.71, iron 4.04. It is found that potassium (71.5 wt. %) predominates among other alkali metals.

Estimation of K_2CO_3 in the ash

A weighed amount (0.2 g) of ash was reacted with dil HCl (0.11 M) as per the reaction:

$$K_2CO_3 + 2\ HCl \rightarrow 2\ KCl + CO_2 + H_2O$$

The filtrate containing unreacted HCl was titrated against standard alkali to estimate K_2CO_3 in the ash. K_2CO_3 in the ash was found to be 0.0304 g (15.2 wt. %).

Effect of ashing temperature on ash produced

Coffee husk was heated in a temperature controlled furnace (Muffle furnace). Known amount of husk (1 g) was heated in a crucible for 15 minutes, at temperatures of 400, 500, 600, 700, 800 and 1000^oC, and the ash obtained was weighed. Figure 1 gives a plot of ‘Ash produced (mass %)’ versus ‘ashing temperature’. Maximum ash yield of 18% was at ashing temperature of 400-500^oC. With the rise in the furnace temperature beyond 500^oC, the amount of ash decreased as ashing became more and more complete. The ash obtained at lower temperatures was blackish in colour, indicating incomplete combustion due to unburned carbon. At higher temperatures more and more of carbon is oxidised to carbon dioxide. As the temperature rises beyond 700^oC, reduction in mass is also due to disintegration of solid compounds, alkali metal salts, into gaseous components.

Effect of ashing temperature on leaching

Samples of ash obtained at various temperatures were leached with deionized water. 100 mL of water was added to 1 g of ash and was stirred for 10 mins, at 20^oC. Residue left after filtration was dried at 90^oC. Percentage of ash leached is given by:

$$\text{Ash leached} = \frac{(\text{Initial mass of ash}) - (\text{Mass of residue after leaching})}{\text{Initial mass of ash}} \text{ x } 100\%$$

Figure 2 shows the 'ash leached, mass%' versus the 'ashing temperature'. Ash leached initially rises with temperature, reaches a maximum value of 73% at 600°C and then drops. The percent leached at 1000°C is 60%. The leachable component of the ash initially rises with temperature because of more complete combustion at higher temperatures. However at temperatures higher than 600°C, the amount of leachables begin to decline. According to Akhmentov, potassium compounds tend to disintegrate and evaporate at temperatures above 700°C, leading to formation of insoluble salts. Also, sintering and partial melting begins at 700°C and decomposition to oxides takes place at 800°C[6].

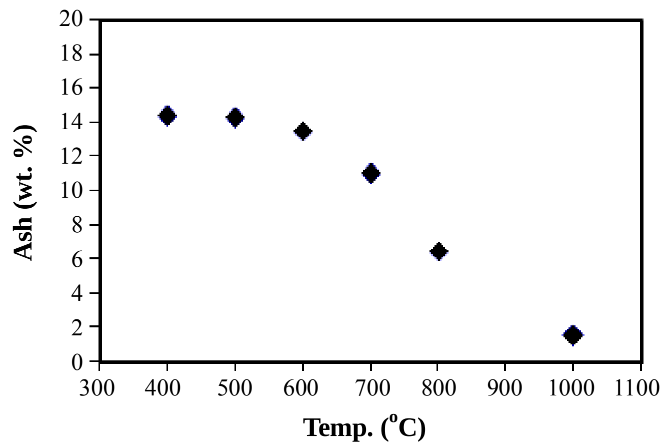

Fig 1: Effect of ashing temp. on ash yield

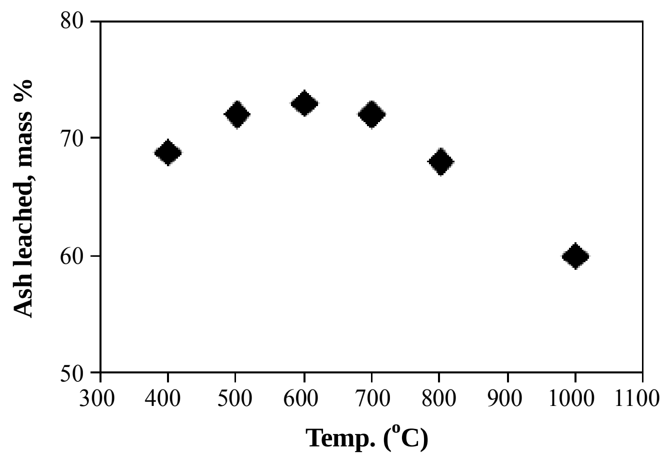

Fig 2: Effect of ashing temp. on ash leached

Effect of stirring on yield

30 g of ash was mixed with 700 mL of deionized water and stirred for 1 minute at 21°C at varying stirring speeds. Conductivity of solution gave an indication of dissolved alkali (yield), Figure 3. It shows that at high stirring speed (above 100 rpm), the solution concentration is almost constant, and therefore stirring has no effect on the leaching.

Effect of ashing temperature, ash particle size, and solvent temperature on the yield of alkali

Effect of the above three variables were studied by a 2^3 Factorial design with centre point. Ash obtained at 400, 650, and 900°C; was sieve analyze for particle size. Ash particles were categorized into 'small' having size range of 40-200 μm, 'medium' having a size range of 200-355 μm, and 'large' having a size range of 355-500 μm. Solvent water temperatures were 20, 40, 55°C. The experiment design with the above three variables (X1, X2, X3) had coded levels of -1, 0, 1; and consisted of 12 runs conducted in duplicate.

Ashing temp., °C (X1): 400, 650, 900 (Coded values -1, 0, 1)

Particle size (X2): Small, medium, large (Coded values -1, 0, 1)

Solvent temp, °C (X3): 25, 40, 55 (Coded values -1, 0, 1)

The yield (Y) of alkali obtained through titration was based on the total alkali in the ash. Polymath 6.10® was used for data analysis and a second order quadratic model was obtained:

$$Y = a0 + a1\ X1 + a2\ X2 + a3\ X3 + a12\ X1.X2 + a13\ X1.X3 + a23\ X2.X3 + a11\ X1^2 + a22\ X2^2 + a33\ X3^2 + a123\ X1.X2.X3$$

The values of parameters obtained are:

a0 = 84.05714, a1 = 1.485714, a2 = -6.457143, a3 = 5.221429, a12 = -0.75, a13 = 0.7214, a23 = 1.22, a11 = -13.328, a23 = -0.714, a33 = -4.2714, a123 = 11.157

It is observed that a12, a13, a23 are small and can be neglected. A revised model after dropping these coefficients is:

$$Y = 83.56028 + 1.159574X1 - 6.4468049\ X2 + 4.379433X3 - 13.47872\ X1^2 - 4.343972X3^2 + 9.723404\ X1.X2.X3.$$

Significance of regression as given by $R^2 = 0.988$, and $R^2_{adj} = 0.975$, shows that the model is satisfactory.

A response surface plot for the yield as a function of (Solvent temperature and Ashing temperature, Figure 4), (Particle size and Ashing temperature, Figure 5), and (Solvent temperature and Particle size, Figure 6) indicate that the 'maximum' yield corresponds to: Ashing temperature of about 600^{o}C, smallest particle size, and highest solvent temperature. Solubility of alkali in water increases considerably with temperature[7], which explains the effect of solvent temperature. Small particle size obviously reduces mass resistance. Also, as mentioned earlier, at ashing temperature higher than 600^{o}C, amount of leachables begin to decline.

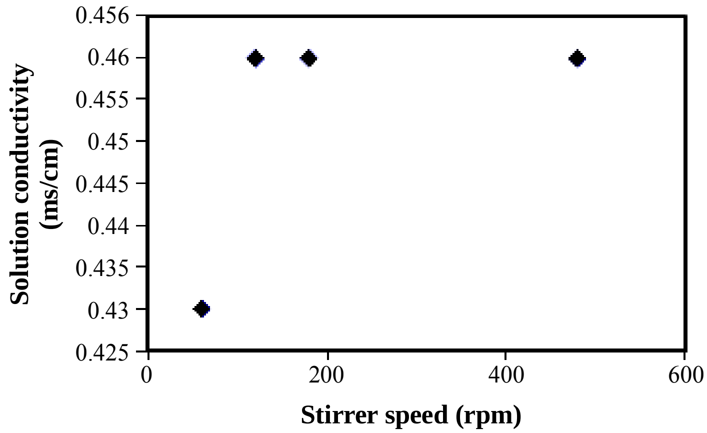

Fig. 3: Effect of stirring on yield

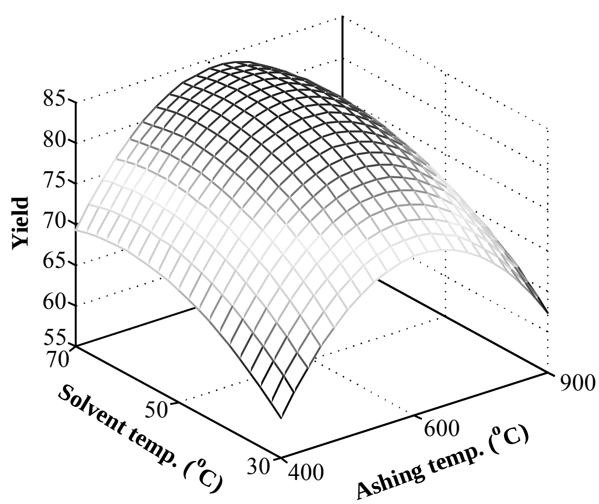

Fig. 4: Response surface plot of yield as a function of solvent temperature and ashing temp.

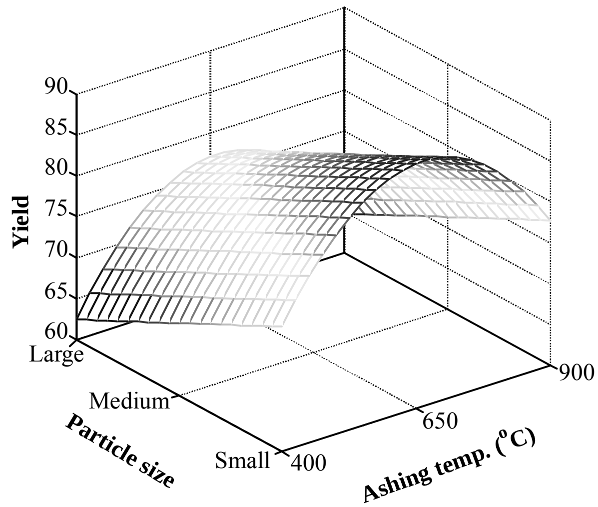

Fig. 5: Response surface plot of yield as a function of particle size and ashing temp.

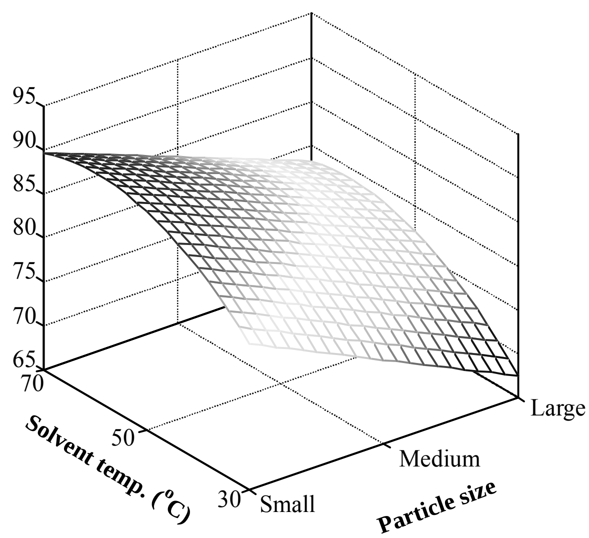

Fig. 6: Response surface plot of yield as a function of particle size and solvent temp.

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