

Article

Comparative Study of the Effect of Temperature on the Adsorption of Metallic Soaps of Shea Butter, Castor and Rubber Seed Oil onto Hematite

J.E. Asuquo^{1, *}, A.C.I. Anusiem², and E.E. Etim¹

¹ Department of Chemistry, University of Uyo, Uyo, Nigeria

² Department of Pure and Industrial Chemistry, University of Port Harcourt, Nigeria

* Author to whom correspondence should be addressed; E-Mail: asuquoje@yahoo.com.

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Abstract: The effect of temperature on the adsorption of potassium, calcium and aluminum soaps of shea butter, castor and rubber seed oils onto hematite in aqueous media was studied. The results showed a decrease in adsorption density with increase in temperature to a minimum at 50 °C for the various soaps. This is attributed to the weakening of the attractive forces between adsorbate and the adsorbent. Adsorption equilibrium was attained between 60 and 70 °C. The maximum adsorption density for the adsorbates is in the order: potassium > calcium > aluminum soaps of all the soaps, which is in the reverse of the size to charge ratio of the cations due to increase in diffusion rate of smaller ions to the surface of the adsorbent. The results also showed that the extent of adsorption is in the order castor oil soap > shea butter oil soap > rubber seed oil soap. This is as a result of the differences in the predominant fatty acids composition of these oils. The level of adsorption of all the adsorbates on the surface of the adsorbent suggests that all the metallic soaps in this study show reasonable degree of surface coverage that may warrant their use as collector reagents in flotation separation of hematite from its ore.

Keywords: adsorption; temperature; metallic soaps; oil; hematite; flotation separation.

1. Introduction

Temperature is a crucial parameter in adsorption reactions. According to the adsorption theory,

adsorption decreases with an increase in temperature and molecules adsorbed earlier on a surface tend to desorb from the surface at elevated temperatures. But for activated carbon, a different trend is noticed where decreasing viscosity and increasing molecular motion at high temperature allows the uptake of molecules into the pores more easily, causing adsorption to increase as temperature increases. However, temperature has not been studied as relevant variable in adsorption experiments. The tests are usually performed at approximately 25 - 30 °C. However, a slight increase in cation uptake by seaweed in the range of 4 to 55 °C has been reported [1]. Adsorption and its phenomena have received a lot of attention by several investigators who reported the dependence of the process on factors such as temperature, pressure, effective surface area, pH, concentration, ionic charge, ionic strength, nature of adsorbate and adsorbent [1-10].

Metallic soaps are metallic salts of fatty acids. They are groups of water-insoluble compounds containing alkaline earth or heavy metals combined with monobasic carboxylic acids of 7 - 22 carbon atoms. They can be represented by the general formula $(RCOO)_xM$, where R is an aliphatic or alicyclic radical and M is a metal of valence X. They differ from toilet soaps in their composition and their insolubility in water. Their solubility or solvation in a variety of organic solvents accounts for their various uses. Soap molecules bear hydrophobic and hydrophilic parts which can be adsorbed selectively on the surface of the desired mineral particles to reduce wetting by water, thereby enhance their flotation. The balance of dynamic forces between the concentration of adsorbate in bulk solution with that at the interface for a particular system leads to adsorption equilibrium [11].

A crude castor oil is a pale straw colour but turns colourless or slightly yellowish after refining and bleaching. The crude oil has distinct odour, but it can easily be deodorized in the refining process. Like any other vegetable oils and animal fats, it is a triglyceride, which chemically is a glycerol molecule with each of its three hydroxyl group esterified with a long chain fatty acid. Its major fatty acid is the unsaturated, hydroxylated 12-hydroxy, 9-octadecenoic acid, known as ricinoleic acid. The fatty acid composition of a typical castor oil contains about 87% of ricinoleic acid [12]. The seeds of rubber tree (*Hevea brasiliensis*) have been found to be rich in oil. Its content in the dried kernel varies from 35 to 45%. It is semi-drying and consists of 17 - 22% saturated fatty acids and 17 - 82% unsaturated fatty acids and is comparable to drying oils commonly used in surface coating. It is composed mainly of linolenic acid (50%) [13]. Shea butter oil botanically called *Butyrospermum parkii* is a soft paste of melted fat with a milky colour in solid form and brownish when melted. It has a characteristic odour. It contains fatty acid triglyceride and a high amount of unsaponifiable matter, which ranges from 2.5 to 15% [14]. Equally, unrefined shea butter oil is superior in that it retains all its natural vitamins, especially vitamins A and E. Crude shea butter has natural antioxidant properties due to its tocopherol content [15]. Shea butter oil has the following fatty acid composition: palmitic acid

(C16) 8.5%, stearic acid (C18) 35.9%, oleic acid (C18) 49.9% and linoleic acid (C18) 5.3% [16].

The effect of temperature on the adsorption of the metallic (potassium, calcium and aluminum) soaps of shea butter, castor and rubber seed oils onto hematite was studied as a prelude in accessing the suitability of using these soaps as collector reagents in ore flotation. Flotation technique is often employed in the separation of solids of similar densities (between 35 and 100 μm) which cannot be separated by gravity using jigging and tabling [17-19]. The objective of this research is to determine the effect of temperature on the adsorption capacity of potassium, calcium and aluminum soaps of shea butter, castor and rubber seed oils on the surface of hematite and to examine the feasibility of using these soaps as collector reagents for the eventual processing of metal ores through the flotation separation technique.

2. Materials and Methods

2.1. Materials

Ore containing samples of hematite obtained from Itakpe, Nigeria (76.9% iron) were crushed in the laboratory using jaw and roll crushers. Gravimetric method of jigging and tabling was employed in removing siliceous materials, while magnetic separation method was used in separating magnetic materials from the ore sample. Sieve analyses were performed using the British standard sieve plates to obtain sample of hematite – 70 micron. Mineralogical analysis of the ore sample was performed with volumetric and spectrophotometric (Buck scientific atomic absorption spectrophotometer - model 205A) methods of analyses. Solutions of reagents were prepared with distilled deionised water, sodium hydroxide and hydrochloric acid solutions were used for pH adjustments. BDH chemical reagents analar grade (of not less than 98% purity) were used in this study. Determination of specific surface area (SSA) was done by the ethylene-glycol-monethyl ether (EGME) method [20].

2.2. Soap Preparation

The 50 g each of shea butter, castor and rubber seed oils was added to each 50 mL of 30% potassium hydroxide, calcium hydroxide and aluminum hydroxide and 30 mL of ethanol into each of the 600 mL beakers, then heated at 90 °C for 1 h in a water bath with vigorous stirring until creamy-pasty soaps are formed. The 50 mL of hot saturated sodium chloride solution was then added to each of the pasty soaps with vigorous stirring for short period and allowed to cool overnight. The soaps cake formed on the surface of the 'lye' were removed and air dried and stored in plastic containers [16].

2.3. Calibration Studies

The electrical conductance of 25 cm^3 soap (potassium, calcium and aluminum soaps of shea

butter, castor and rubber seed oils) solutions of different concentrations were measured at 29 °C and pH 7.31, using an Electronic conductivity meter (model-90 check-mate deluxe field system-corning). From the result, a calibration graph of the soap conductance was plotted against the square root of soap concentrations based on the Kohlrausch law equation [21]:

$$\Lambda_m = \Lambda_m^\circ - KC^{1/2} \dots \dots \dots (1)$$

Where Λ_m is the molar conductivity, Λ_m° is the limiting molar conductivity, C is the concentration of the solution and K is a constant.

3. Results and Discussion

The chemical composition of hematite is given in Table 1. Tables 2, 3 and 4 show the adsorption densities of K^+ , Ca^{2+} and Al^{3+} soaps of shea butter oil onto adsorbent (hematite) at various temperatures. Tables 5 and 6 present data for K^+ and Ca^{2+} castor oil soaps while Tables 7, 8 and 9 represent that for K^+ , Ca^{2+} and Al^{3+} soaps of rubber seed oil. Calculations from experimental results and sample analysis show that the specific surface area (SSA) of hematite is 118.5 m²/g. This value is in agreement with that obtained by Ibezim-Ezeani and Anusiem [22] for hematite using the same method. The plots of the conductance of potassium, calcium and aluminum soaps of shea butter, castor seed and rubber seed oils against the square root of initial soap concentrations were used for subsequent determination of concentration in the various experiments (Figs. 1, 2 and 3).

Table 1. Chemical composition of hematite (Fe₂O₃)

| Chemical Composition | Hematite (%) |
|----------------------|--------------|
| Fe ³⁺ | 76.9 |
| S ²⁻ | 0.1 |
| Al ³⁺ | 4.1 |
| Si ⁴⁺ | 8.3 |
| Mg ²⁺ | 0.4 |
| Ca ²⁺ | 0.3 |
| P ³⁻ | 0.1 |
| Ti ⁴⁺ | 0.2 |
| Mn ²⁺ | 0.1 |

Table 2. K⁺ shea butter oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial K ⁺ soap concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium K ⁺ soap concentration (g/L) | K ⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of K ⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 152 | 4.69 | 5.53 | 0.00466 |
| 40 | 1.0 | 171 | 6.28 | 3.72 | 0.00313 |
| 50 | 1.0 | 179 | 6.96 | 3.04 | 0.00256 |
| 60 | 1.0 | 184 | 7.41 | 2.59 | 0.00218 |
| 70 | 1.0 | 184 | 7.41 | 2.59 | 0.00218 |

Table 3. Ca²⁺ shea butter oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial Ca ²⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium Ca ²⁺ soap Concentration (g/L) | Ca ²⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of Ca ²⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 154 | 4.86 | 5.14 | 0.00433 |
| 40 | 1.0 | 173 | 6.44 | 3.56 | 0.00300 |
| 50 | 1.0 | 182 | 7.20 | 2.80 | 0.00236 |
| 60 | 1.0 | 188 | 7.59 | 2.41 | 0.00203 |
| 70 | 1.0 | 188 | 7.59 | 2.41 | 0.00203 |

Table 4. Al³⁺ shea butter oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial Al ³⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium Al ³⁺ soap Concentration (g/L) | Al ³⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of Al ³⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 177 | 6.74 | 3.26 | 0.00275 |
| 40 | 1.0 | 184 | 7.29 | 2.72 | 0.00228 |
| 50 | 1.0 | 191 | 7.93 | 2.07 | 0.00174 |
| 60 | 1.0 | 199 | 8.53 | 1.47 | 0.00124 |
| 70 | 1.0 | 199 | 8.53 | 1.47 | 0.00124 |

Table 5. K⁺ castor seed oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial K ⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium K ⁺ soap Concentration (g/L) | K ⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of K ⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 58 | 2.07 | 7.93 | 0.00669 |
| 40 | 1.0 | 91 | 5.31 | 4.69 | 0.00395 |
| 50 | 1.0 | 97 | 6.37 | 3.63 | 0.00306 |
| 60 | 1.0 | 103 | 7.23 | 2.77 | 0.00233 |
| 70 | 1.0 | 103 | 7.23 | 2.77 | 0.00233 |

Table 6. Ca²⁺ castor seed oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial Ca ²⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium Ca ²⁺ soap Concentration (g/L) | Ca ²⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of Ca ²⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 63 | 2.53 | 7.47 | 0.00630 |
| 40 | 1.0 | 93 | 5.62 | 4.38 | 0.00369 |
| 50 | 1.0 | 100 | 6.67 | 3.33 | 0.00281 |
| 60 | 1.0 | 104 | 7.44 | 2.56 | 0.00216 |
| 70 | 1.0 | 104 | 7.44 | 2.56 | 0.00216 |

Table 7. K⁺ rubber seed oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial K ⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium K ⁺ soap Concentration (g/L) | K ⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of K ⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 87 | 4.94 | 5.06 | 0.00427 |
| 40 | 1.0 | 103 | 7.23 | 2.77 | 0.00233 |
| 50 | 1.0 | 107 | 7.98 | 2.02 | 0.00170 |
| 60 | 1.0 | 110 | 8.40 | 1.60 | 0.00135 |
| 70 | 1.0 | 110 | 8.40 | 1.60 | 0.00135 |

Table 8. Ca²⁺ rubber seed oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial Ca ²⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium Ca ²⁺ soap Concentration (g/L) | Ca ²⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of Ca ²⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 89 | 4.97 | 5.03 | 0.00424 |
| 40 | 1.0 | 106 | 7.78 | 2.22 | 0.00187 |
| 50 | 1.0 | 113 | 8.60 | 1.40 | 0.00118 |
| 60 | 1.0 | 115 | 8.75 | 1.25 | 0.00105 |
| 70 | 1.0 | 115 | 8.75 | 1.25 | 0.00105 |

Figs. 4, 5 and 6 show the plots of adsorption of these soaps as a function of temperature. The tables and figures above show that the adsorption densities decrease with increasing temperatures to a minimum at 50 °C for the various soaps. The decrease in adsorption density with increasing temperature is likely due to the weakening of attractive forces between adsorbate and the adsorbent. This enhances the tendency of the adsorbate ions to escape from each adsorbent surface to the solution phase, hence desorption may start to occur above this temperature. Also, at high temperature, the thickness of the boundary layer decreases, due to the increased tendency of the metal ion to escape from the biomass surface to the solution phase, which results in a decrease in adsorption as temperature increases [23]. However, between 60 and 70 °C the adsorption density remains constant. This may be attributable to the denaturation or distortion of the adsorbate molecules. This will cause the structured water molecules surrounding the hydrophobic (non-polar) groups to be disrupted [24]. These are likely to alter the orientation and effective interaction at the site. Physical adsorption usually decreases with increase in temperature while chemisorption is favored at higher temperatures [25]. For physical adsorption, we expect that the adsorption will be higher at the lower temperature than at higher temperature whereas if the process is a chemical adsorption, the reverse is true [26]. The observed decrease in adsorption density suggests that the adsorption on the adsorbent is largely a physical process. This is in line with the results obtained by Horsfall and Spiff [1], Ekpe et al. [27] and Fouda et al. [28]. However, generally, the adsorption theory suggests that adsorption decreases with an

increase in temperature, and molecules adsorbed earlier on a surface tend to desorb at elevated temperatures [29].

Table 9. Al³⁺ rubber seed oil soap adsorption density at different temperatures for hematite

| Temperature (°C) | Initial Al ³⁺ soap Concentration (g/L) | Equilibrium conductance (μS/cm) | Equilibrium Al ³⁺ soap Concentration (g/L) | Al ³⁺ soap adsorbed per gram of hematite (g/g) | Adsorption density of Al ³⁺ soap per m ² hematite surface (g/m ²) |
|------------------|---|---------------------------------|---|---|---|
| 29 | 1.0 | 90 | 4.99 | 5.01 | 0.00422 |
| 40 | 1.0 | 108 | 7.69 | 2.31 | 0.00194 |
| 50 | 1.0 | 114 | 8.62 | 1.38 | 0.00116 |
| 60 | 1.0 | 116 | 8.77 | 1.23 | 0.00101 |
| 70 | 1.0 | 116 | 8.77 | 1.23 | 0.00101 |

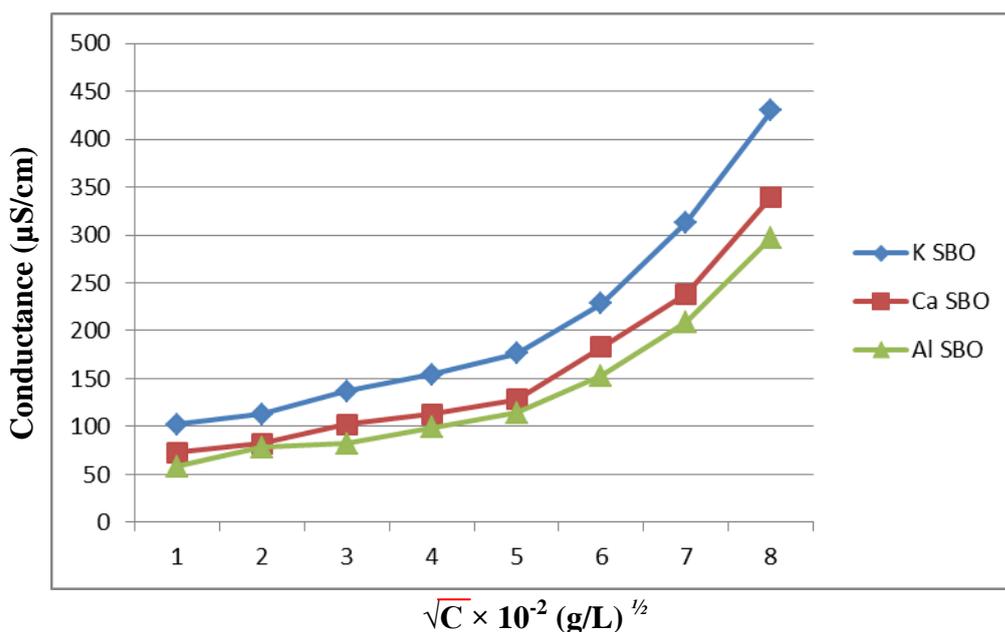


Figure 1. Plot of conductance versus concentration for K⁺, Ca²⁺ and Al³⁺ shea butter oil soaps.

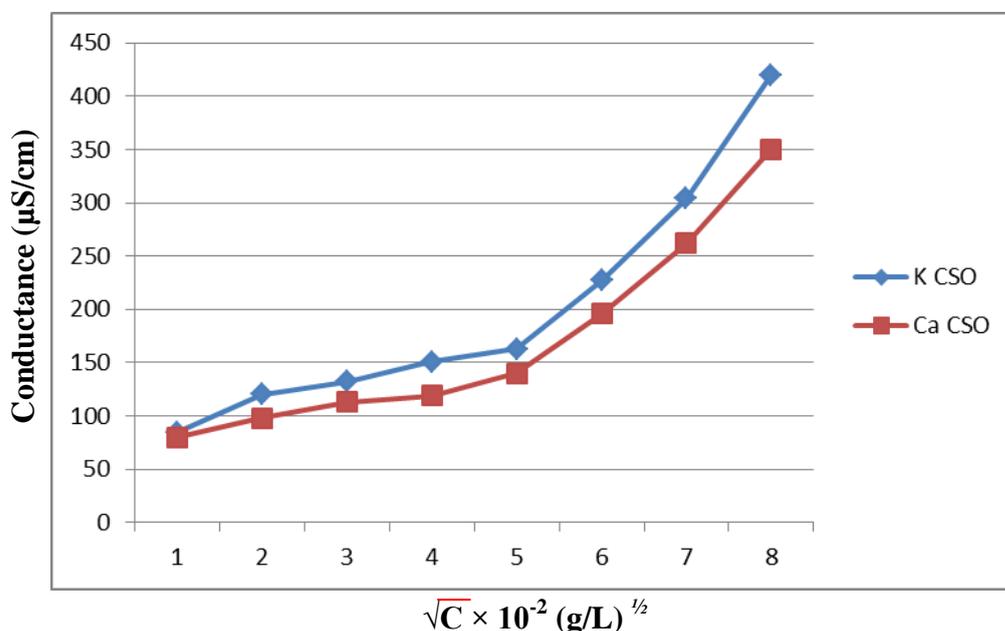


Figure 2. Plot of conductance versus concentration for K⁺, Ca²⁺ and Al³⁺ castor seed oil soaps.

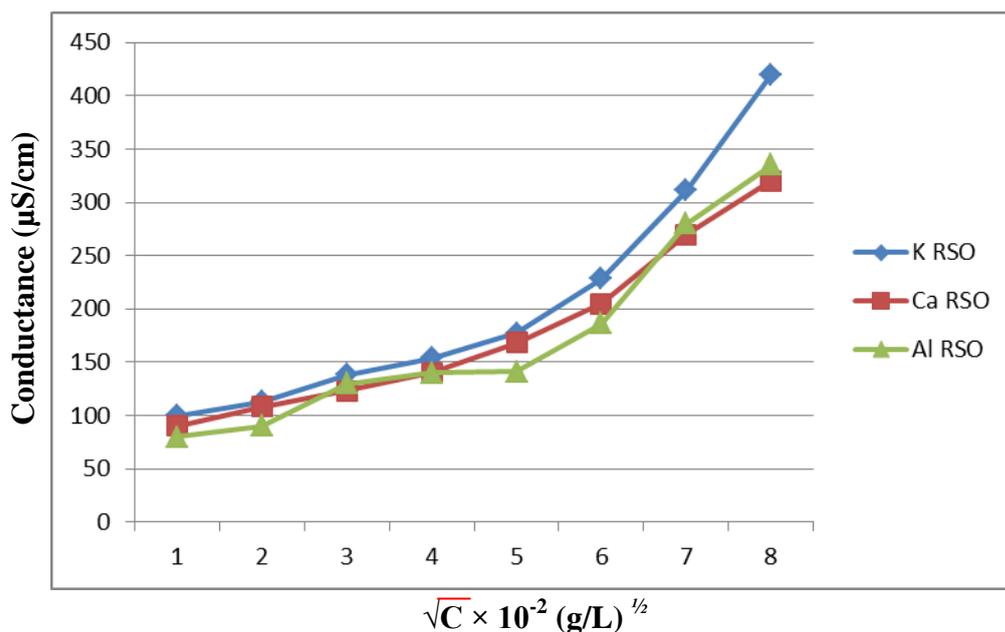


Figure 3. Plot of conductance versus concentration for K^+ , Ca^{2+} and Al^{3+} rubber seed oil soaps.

The adsorption density shows the highest values for K^+ castor oil soap followed by that of Ca^{2+} castor oil soap at the same temperature. The adsorption densities of all the K^+ soaps investigated in this study showed that the soap of castor oil has the highest adsorption density followed by shea butter oil soap, then lastly by rubber seed oil soap of the same metal. This sequence is common among all soaps of the various ions. From the foregoing, it is likely that these differences in adsorption densities may arise from the differences in the predominant fatty acids composition of these oils. Castor oil is predominantly composed of ricinoleic acid (90%), an unsaturated fatty acid, with a hydroxyl group while shea butter oil is predominantly composed of oleic acid (50%) and rubber seed oil is composed of linolenic acid (50%). These subtle differences in the fatty acids between these oils are likely reflected to a certain degree in their adsorption while the presence of OH^- group in the fatty acid which is predominant in castor oil may have a far more reaching effect in influencing the adsorption density through electrostatic effects than the unsaturation.

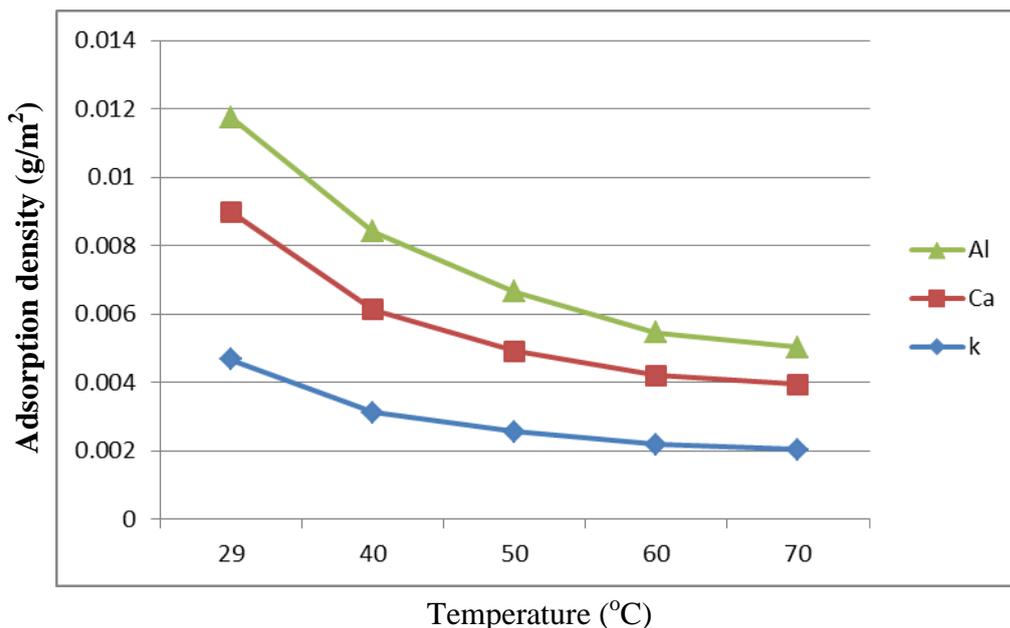


Figure 4. Plot of adsorption density at different temperatures for K^+ , Ca^{2+} and Al^{3+} for shea butter oil soaps.

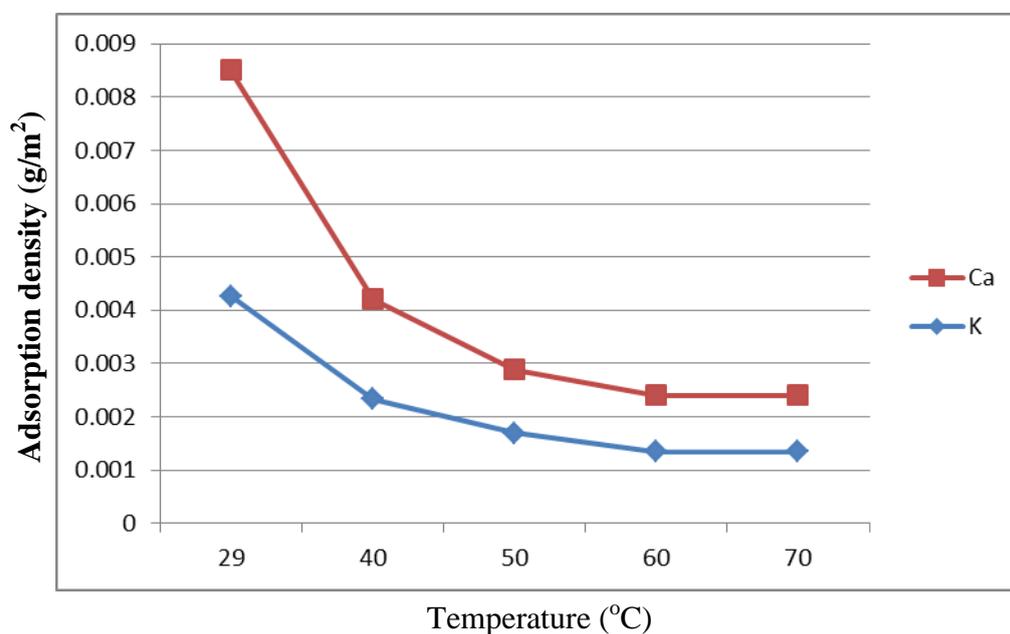


Figure 5. Plot of adsorption density at different temperatures for K^+ and Ca^{2+} for castor seed oil soaps.

Considering the various soaps of shea butter oil, potassium soap has the highest adsorption density followed by calcium soap and lastly by aluminum soap. This indicates that there is a correlation between the adsorption density and the size to charge ratio of the cations which are 0.75, 2.02 and 5.77 for potassium, calcium and aluminum, respectively. This trend is also observed for all the soaps of castor oil and rubber seed oil. The likely origin of these differences may be related to increased diffusion rate of the smaller ion to the surface of the adsorbent.

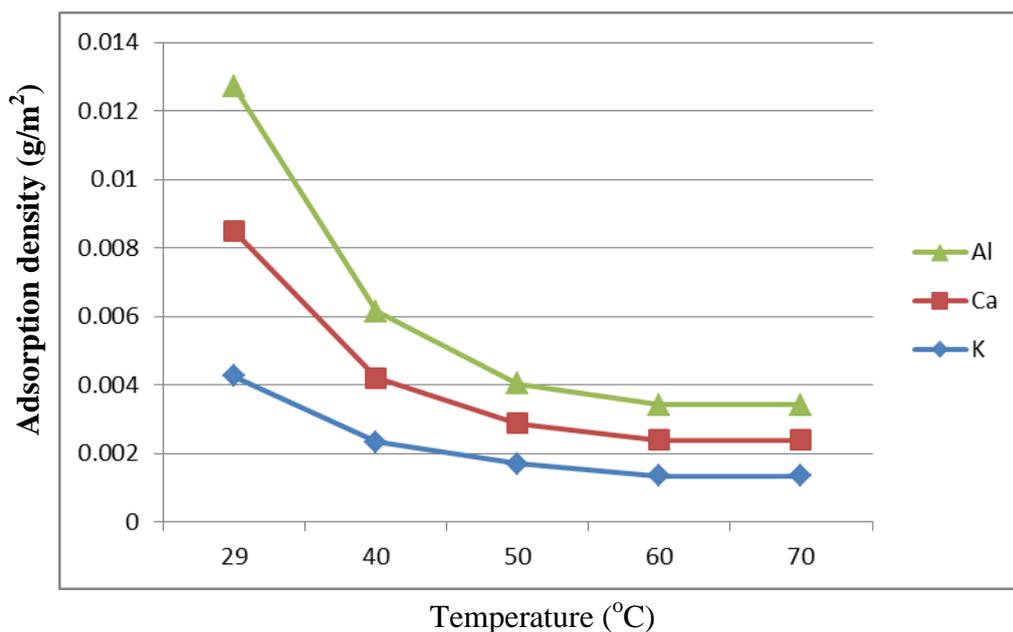


Figure 6. Plot of adsorption density at different temperatures for K^+ , Ca^{2+} and Al^{3+} for rubber seed oil soaps.

4. Conclusions

This investigation has shown the effect of temperature on the adsorption of potassium, calcium and aluminum soaps of shea butter, castor and rubber seed oils onto hematite. The results showed that the adsorption densities decrease with increasing temperatures to a minimum at 50 °C for the various soaps. The decrease in adsorption density with increasing temperature is likely due to the weakening of attractive forces between adsorbate and the adsorbent. Adsorption equilibrium was attained between 60 and 70 °C. The maximum adsorption capacity for the adsorbates is in the order potassium soap > calcium soap > aluminum soap of shea butter oil, which is in the reverse of the size to charge ratio of the cations due to increased diffusion rate of smaller ion to the surface of the adsorbent. The results also showed that the extent of adsorption is in the order castor oil soap > shea butter oil soap > rubber seed oil soap. This is as a result of the differences in the predominant fatty acids composition of these oils. The level of adsorption of all the adsorbates on the surface of the adsorbent suggests that all the metallic soaps in this study show reasonable degree of surface coverage that may warrant their use as collector reagents in flotation separation of hematite from its ore.

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