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CASE STUDY

Studies on the use of locally manufactured soaps as alternative collectors for the flotation of iron ores

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Abstract
Industrially approved collectors are known to be expensive in the mining sector, which raises production costs. This research shows how three locally manufactured soaps can be used as an alternative for collectors in the flotation process. The chemical composition of the iron oxide sample was determined using X-ray fluorescence (XRF). The soaps were graded according to total fatty matter, free caustic alkali, and lather volume to demonstrate their quality. The iron oxides, which made up 67 % of the total weight, were floated with naphthalene as a frother and locally manufactured soaps, namely Key soap, Alata soap, and Azumah Blow soap, at a collector dosage of 0.6 ml, 1 ml, and 1.4 ml. This was done to establish which soap is the most efficient collector and the dosage that results in the best recovery. Atomic Absorption Spectrometry (AAS) was used to determine the iron content that reported in the feed, concentrates and tailings. Optimum collector dosage was 1 ml, with Alata soap generating the highest recovery of 70.5 %, which compares well with conventional collectors used in iron oxide flotation. The studies also revealed that the total fatty matter, free caustic alkali, and lather volume of locally made soaps have a positive influence on the flotation of iron oxide. However, only Key soap met all the specifications for soaps by the Ghana Standards Authority.

Keywords: Collector, Soap, Recovery, Total Fatty Matter, Free Caustic Alkali, Lather Volume

Introduction
Froth flotation uses the difference in physicochemical surface properties of minerals to achieve specific separations from ores (Wills and Finch, 2015). Historically, it was first used in the mining industry, where it was one of the great enabling technologies of the 20th century (Ma, 2012). Its development has improved the recovery of valuable minerals, such as gold, iron, copper, and lead-bearing minerals. The process is widely accepted due to its accuracy and higher recovery of minerals of interest from much lower-grade ores (Jyoti *et al.*, 2010). The process selectively separates the mineral of interest from its gangue by taking advantage of differences in its hydrophobicity and hydrophilicity. Flotation of mineral particles however does not depend only on the hydrodynamics, but also the appropriate selection of different reagent combinations. Reagents are mainly used for treating the surface of ores and improving conditions necessary for flotation efficiency. These reagents impact the pulp chemistry hence making the froth a chemical complex. The flotation agents may remain in the wastewater since they are not biodegradable. They end up polluting the mining environment. They also have an impact on downstream processes. Reagents may be collectors, depressants, dispersants, and frothers of which the collector plays a major role (Nakhaei and Irannajad, 2018).

In the froth flotation of iron oxide ores, many different collectors are used to make the mineral of interest hydrophobic or hydrophilic. Mixed collectors in iron ores flotation exhibited superior results (Vidyadhar *et al.,* 2012). This collector combination comes at a very high cost; therefore, most industries spend more in acquiring these collectors. The net profit made by these industries becomes minimal. Also, sodium oleate a commonly used collector in iron ores is recorded to be efficient in the acidic pH range (Bhadani *et al.*, 2017). These disadvantages make the issue of collector in iron oxide a concern. Meanwhile, the bulk of industrially used collector chemistries are classified as soaps, and they have much of the same characteristics as all surfactants (Michaud, 2015), making soaps an alternative worth investigating.

Kent (2003) technically defined soap as the alkali salt of a fatty acid. It is the product that results from the reaction of fatty acid and a strong base (alkali). According to Katz (2000), a soap molecule consists of a long hydrocarbon chain (composed of carbons and hydrogen) with a carboxylic acid group on one end which is ionically bonded to a metal ion, usually sodium or potassium. The hydrocarbon end is nonpolar and is soluble in nonpolar substances (such as fats and oils), and the ionic end (the salt of a carboxylic acid) is soluble in water.

Soap Manufacturing has increased in recent times and as a result, many industries have been established in Ghana (Amenumey, 2008). Examples of soap produced by these companies include Key soap, sunlight washing soap, Brillant, guardian, propa soap, among others. There are also small-scale industries that produce soaps for washing and bathing. Notable soaps among these are “alata” soap, “amonkye” soap, “Azumah Blow” soap, “ameenshangari” soap, etc. These soaps are mostly used by rural and peri-urban dwellers (Kwame, 2012).

The large factories in Ghana use standardized processes (uniform quantity of ingredients, time for mixing, temperature control, etc.) in their soap production. They develop their products based on sound scientific research and technological data, which conform to the Ghana Standards Authority’s specifications as well as international standards (Baffoe and Matsuda, 2018). On the contrary, it is perceived that small-scale soap producers use non-standardized processes in their soap manufacturing often leading to noticeable levels of product variations. The difference in the manufacturing processes of the soaps is likely to have different effects on recovery when subjected to flotation (Rosen and Kunjappu, 2012). In this work, therefore, the conformity of locally manufactured soaps to the approved standards was assessed and their efficiency as collectors is also investigated.

Materials and Methods
Sample preparation
Soap
The three brands of soap used in this study; Key soap (by Unilever Ghana Limited) and both Alata soap and Azumah Blow produced by small-scale manufacturers were purchased from Kumasi (Tech-junction). These three soap brands were selected because they are popular, least expensive, and widely available on the Ghanaian market. Attrition with a plastic grater was used to break the soap into shreds. This was done to reduce

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the particle size and increase the soap's surface area hence increasing its reactivity (Sohn and Wadsworth, 2013).

Ore

Iron oxide ore samples were obtained from a mine in Tarkwa, Ghana. The total sample taken was 5 kg. The ore samples were kept in sample bags for subsequent laboratory work. The ore was crushed using a jaw crusher (KHD Humboldt Wedag AG). The crushed ore was fed into a laboratory ball mill (15 kg charge steel balls, 73 rev/min), where the particle size was further reduced to liberate the desired mineral from the ore (Perez-Johnson and Campbell, 2015). It was then pulverized to 200 microns. A Test Sieve Shaker was used to achieve screen analyses of ground materials. The sample was placed in an oven and dried at 105 ℃ for 90 minutes.

Characterization of soaps

Total fatty matter

Five grams of soap was weighed and transferred into a 250 ml beaker. A hundred (100) ml of warm water (50 ^{o}C) was added to completely dissolve the soap. Forty (40) ml of 0.5 M HNO_3 was added to the content. The mixture was heated over a water bath until fatty acids floated as a layer above the solution. The solution was then cooled suddenly in ice water to solidify the fatty acids and was subsequently separated. Fifty (50) ml of chloroform was added to the remaining solution and transferred to a separating funnel. The solution was then shaken and allowed to separate into two layers. The bottom layer was drained and 50ml of chloroform was added to the remaining solution in the separating funnel. The dissolved in the chloroform was separated and transferred to the collected fatty matter. The fatty matter was then weighed, the content allowed to evaporate, and the residue weighed. From the difference in weight, the % of the fatty matter in the given sample was determined.

Alkalinity

Ten grams of the soap sample was weighed into a clean dry Erlenmeyer flask and dissolved in 50 ml of distilled water. Three drops of methyl orange indicator were added to the solution and titrated against 1 M HCl solution till the colour changed from orange to pink. The procedure was repeated thrice to get consistent titres and the value averaged and calculated.

Free caustic alkali (FCA)

Five grams of soap were dissolved in 100 ml of neutralized ethanol over a steam bath and 10 ml of barium chloride was added to the hot solution. The soap sample was titrated against 0.1 M H_2SO_4, using a phenolphthalein indicator. The amount of free caustic alkali in the soap is calculated using the Eqn. (1) by Beetseh *et al*. (2013) given as:

$$FCA = \text{molarity of acid x formula weight of barium acid used} \quad (1)$$

pH

One gram of the soap was measured and added to 9.0 g of water. The soap was allowed to dissolve in the water for about five minutes. The pH values were then determined using the pH-012 model.

Lather volume

Ten grams of the soap was added to 100 ml of distilled water in a measuring cylinder. The mixture was then shaken vigorously to generate foam. After shaking for about 90 seconds, the measuring cylinder was allowed to stand for 150 seconds. The height of the foam in the solution was then measured and recorded.

Flotation process

The flotation reagent suite included a soap sample as a collector (1 % soap solution), and 18 % Nitrogen sulphide as frother. An impeller speed of 1100 rev/min was maintained for conditioning at 20 % solids by weight of the pulp. Flotation was conducted at a pulp pH between 7 – 7.5. For each soap, collector dosages of 0.6 ml, 1.0 ml, and 1.4 ml were used. The oxygen flow rate was 4–5 L/min respectively. Flotation tests were conducted in a Denver-D12 laboratory flotation machine and a 1-litre vessel. The froth was collected for 5 minutes. The froth was collected in a tray and dried.

Iron recovery

The collected froth after drying was weighed and both the mass of concentrates and tailings were measured and recorded. Five grams of the sample was digested using HCl and HNO_3 in a ratio of 3:1 respectively (Elwood, 2021). The solution with the sample was agitated using a magnetic stirrer for 45 minutes to ensure effective leaching and the solution and filtered. The filtrate was analyzed using AAS. The iron recovery was computed using Eqn. (2) by Wills and Finch (2015) given as:

$$Iron\ Recovery(\%) = \frac{100c(f-t)}{f(c-t)} \quad (2)$$

Where c = the grade (assay) of iron in the concentrate; f = grade of iron in the feed and t = the grade of iron in tails.

X-ray fluorescence spectrometry

For powdered samples, about 4 g of the sample and 0.9 g of a binder are weighed and homogenized using a mill. The mixture is then poured into the die and pressed with 15 tons to a 32 mm pellet using the hydraulic press. The pellet is then kept in a sample holder for XRF analysis. For samples that do not require binder because of the type of elements to be analyzed or the type of method on the analyzer, they are pressed into the pre-flared 31 mm spec cap or rubber sample cups covered with XRF thin film before analysis. Pulverized organic samples fall under this category. Samples such as metal ingots are analyzed directly without pulverization. Produced organic foods that are homogenous in nature and so do not need pulverization are also analyzed directly. Liquid or molten samples are poured into XRF analysis cups and covered with XRF thin film before analysis.

In XRF, an electron from one of the atom's inner orbital shells gets expelled when an X-ray with enough energy (higher than the atom's K or L shell binding energy) strikes an atom in the sample. When the atom stabilizes again, an electron from one of its higher shells enters the vacancy in the inner shell. Through the emission of a fluorescent X-ray, the electron transitions to a lower energy state. This X-ray's energy is equivalent to the precise energy differential between the electron's two quantum states. The foundation of XRF analysis is the measurement of this energy (Schramm, 2016).

Atomic absorption spectroscopy

An Atomic Absorption Spectrometer (AAS) was filled with 10 ml of liquified concentrate and tailings. By applying distinctive electromagnetic radiation wavelengths from a light source, it was able to identify elements. Different elements absorbed wavelengths in different ways, and standards were used to measure these absorbances.

Analytes were initially atomized in AAS in order to record and emit their distinctive wavelengths. Subsequently, as atoms absorbed a particular amount of energy during excitation, electrons in those atoms advanced one energy level. This energy was associated with a certain wavelength that defines the element. It is possible to identify particular elements and determine their concentrations based on the wavelength and intensity of the light (Nielsen et al., 2010).

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Results and Discussion

Observation

From the samples, the colour of the Key soap was yellow, Alata soap was dark brown and Azumah Blow soap was cream in colour. The lather volume test conducted showed how differently the soaps lathered with water. Key soap created the most lather followed by Alata and then Azumah Blow soap. Initially, Azumah Blow soap yielded the most lather however over 2-5 minutes, the lather began to break. This is because the lather formed was weak and breaks down. Key soap had a very strong and stable froth. Alata soap also has a strong and stable froth however it does not lather easily when compared to that of Key soap.

Upon adding the barium chloride solution, the samples turned creamy and then turned pink upon adding the indicator. The creamy change symbolizes the precipitation of the fatty oils; so, they do not interfere with the chemistry of the process.

Characteristics of soaps

According to Antonić *et al.* (2020), the chemical properties of soaps highly influence their effectiveness. Similarly, the chemical characteristics of soaps affect the hydrophobicity, contact angle, metal selectivity, and recovery among others. Thus, different chemical characteristics of soaps are likely to have different impacts on the flotation process.

The basic chemical characteristics of Key soap, Alata soap and Azumah Blow soap were therefore determined to explain its effect on hematite during its flotation process. The parameters were: Net Weight (g), free caustic alkali (NaOH) (%), lather volume (ml), total fatty matter (%), and pH. The standard values were obtained from the Ghana Standards Authority, Accra, and are presented in Table 1. The mean values for Key soap parameters were net weight (1073 g), lather volume (360), total fatty matter (36.25), free caustic alkali (0.062), and pH (10.61). The mean values for Azumah Blow soap parameters were net weight (341.6 g), free caustic alkali (0.13), lather volume (240), total fatty matter (27.92), and pH (11.89). The values obtained for Alata soap were net weight (370 g), free caustic alkali (0.15), lather volume (260), total fatty matter (38.33), and pH (10.05).

The deviation of the Azumah Blow and Alata soap from the recommended
standard specifications of laundry soaps (GS 167: 2006) could be attributed to the non-standardized process in its preparation, the scale of production, and the technology used. The values of the basic chemical characteristics (parameters) of Key, Azumah Blow, and Alata soaps varied from each other. The implication is that all soaps are likely to have different impacts on the flotation of hematite. According to Atkins and Atkins (2003), small-scale soap producers develop their methods of making the soap as well as selecting raw materials in sincere ignorance, without thinking of safety measures that can compromise the quality of the soaps produced. Such methods of production mostly result in either too much alkali or too much oil or fats being used during the soap preparation. Atkins and Atkins (2003) concluded that the difference in the manufacturing processes of the soaps is likely to have different effects on their cleansing mechanism when subjected to washing. Hence due to the differences in the chemical properties of the three soaps, they were expected to influence the flotation process differently.

Effects of the chemical characteristics of coaps on flotation

The pH of the soap affects the nature, and the size of the bubble formed. The soap samples recorded pH values of 10.61, 10.05 and 11.89 for Key soap, Alata soap and Azumah Blow soap respectively. Table 2 shows the relationship between the pH of the surfactant and the nature of the bubble formed.

From the Table 2, it can be inferred that the pH influences the bubble size and the froth characteristics. Alata soap and Key soap have relatively the highest bubble size of 2-3 mm with a dense bubble swarm, with Azumah Blow soap recording that of 1-2 mm with a very dense bubble swarm froth. Between pH 10-11, flotation of iron oxides using sodium oleate produces dense bubble swarm of size 2-3 mm (Quast, 2017).

Key soap had a total fatty matter of 36.25 %, Alata soap 38.33 %, and Azumah Blow soap 27.92 %. The total fatty matter increases the contact angle by increasing the interaction and adsorption between the mineral surface and the collector. This, subsequently, increases the hydrophobicity of the mineral enabling it to float to the surface easily.

The higher the FCA, the more alkali the soap. Alkalis raise the pH of the pulp, which assists in breaking up oily and acidic components to enhance adsorption. Alata soap recorded the highest FCA of 0.15, followed by Azumah Blow at 0.13 and then Key soap at 0.06.

The results obtained showed that Key soap produced the highest lather at 360 ml, followed by Alata soap at 260 ml and then Azumah Blow soap at 240 ml. The lather volume significantly reveals the ability of the bubbles created to hold the particle. In froth flotation lather volume plays a crucial role in creating a stable froth and consequently selectivity and recovery of the process. Froth with very high stability is not desired in flotation because though it favours flotation of

Table 1 Basic chemical characteristics of the locally made soaps

Parameter	Azumah Blow	Key soap	Alata soap	Standard Specification for Soap by GSA (GS 167:2006)
Net weight	341.6	1073	370	1200
Free Caustic Alkali (%)	0.13	0.062	0.15	0.1 (max)
Lather Volume (ml)	240	360	260	200.0 (min)
Total Fatty Matter (%)	27.92	36.25	38.33	59.0 (min)
pH	11.89	10.61	10.05	-

Table 2 pH of surfactant relation with nature of the bubble formed during flotation of hematite (Quast, 2017)

Soap	pH	Approximate dominant bubble size (mm)	Bubble and froth characteristics
Azumah Blow	11.89	1-2	Very dense bubble swarm, froth at the air-water interface
Key soap	10.61	2-3	Dense bubble swarm
Alata Soap	10.05	2-3	Dense bubble swarm

valuable minerals it also enhances particle entrainment which can in turn reduce recovery (Ostadrahimi *et al.,* 2021).

Chemical composition of iron ore

Table 3 shows the chemical composition of the iron oxide ore. The result shows the presence of two major oxides: Fe_2O_3 (66.74 %) and Al_2O_3 (16.0 %). Other minor oxides like MgO, TiO_2 and SiO_2 were also found to be present.

Table 3 Chemical composition of the iron oxide ore

Major Oxides	Composition (%)
MgO	1.0
Al_2O_3	16.0
SiO_2	1.0
P_2O_5	0.1
SO_3	0.2
CaO	0.1
TiO_2	1.0
MnO_2	0.01
Fe_2O_3	66.74

Figure 1 shows the recoveries obtained after the flotation process. Alata soap performed well in recovering iron oxide followed by Key soap and then Azumah Blow. This result may be attributed to the relatively higher value of total fatty matter which enabled the formation of a higher contact angle. Alata soap showed a decreasing order with an increase in dosage which may be associated with higher dosages floating both iron oxide and gangue. For Key soap and Azumah Blow, however, a dosage of 1 ml gave the higher recoveries, indicating the possibility of collector dosage not being enough to float the ore while a dosage of 1.4 ml floated both mineral of interest and gangue.

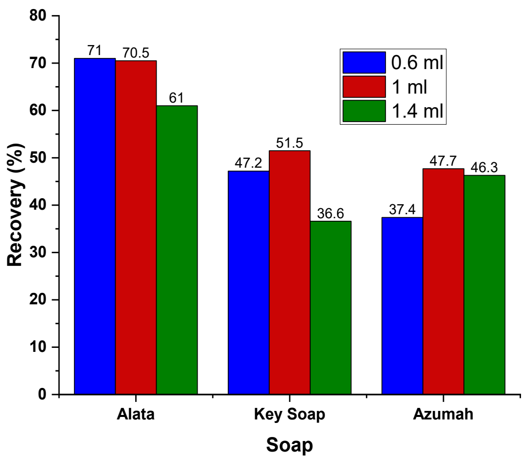

Figure 1 Percent recovery of iron

Conclusions

In this research characterization of 3 locally manufactured soaps and their efficiencies as collectors were conducted. The soaps were characterized using pH, alkalinity, Free Caustic Alkali, Total Fatty Matter and Lather Volume. From the result obtained it can be concluded that; when compared to the Ghana Standard Authority specification, Azumah Blow soap and Alata soap were confirmed as non-standardized, while Key soap on the other hand met the specifications. The iron oxide ore is composed of 67 % iron oxide (Fe_2O_3) and hence is considered a high-grade ore.

All soaps worked efficiently as collectors and Alata soap had the highest recovery. Optimum collector dosage is 1 ml. This is because Alata soap at a dosage of 1 ml has a recovery of 70.5 %, which compares with the highest recovery (71.00 %) achieved at 0.6 ml. Additionally, for the other two soaps (Key soap and Azumah Blow soap) both had their highest recovery values of 51.5 % and 47.7 % respectively at 1.0 ml.

Conflict of Interest Declarations

Authors declare that there is no conflict of interest.

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