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**Article** *in* International Journal of Engineering Research in Africa · June 2020

DOI: 10.4028/www.scientific.net/JERA.49.152



**7 authors**, including:

Petrus Nzerem
Nile University of Nigeria


Oghenerume Ogolo
Nile University of Nigeria


Ikechukwu Stanley Okafor
Nile University of Nigeria


A. M. Gimba
Gombe state politechnic Bajoga

*International Journal of Engineering Research in Africa*
*ISSN: 1663-4144, Vol. 49, pp 152-172*
*doi:10.4028/www.scientific.net/JERA.49.152*

*Submitted: 2019-08-24*
*Revised: 2020-03-20*
*Accepted: 2020-03-24*
*Online: 2020-06-29*

# Study of the Potential of Sodium Carbonate Extracted From Trona as a Drilling Fluid Additive

Petrus Nzerem[1,a], Enyo June Adejoh[1,b], Oghenerume Ogolo[1,c*], Ikechukwu Okafor[1,d], Abdullahi Suleiman Bah Gimba[1,e], Ternenge Joseph Choir[1,f], Precious Ogbeiwi[2,g]

[1]Department of Petroleum and Gas Engineering, Nile University of Nigeria, Abuja, Nigeria.

[2]Institute of GeoEnergy Engineering, Heriot-Watt University, Edinburgh, UK

[a]petrus.nzerem@nileuniversity.edu.ng, [b]aenyojune@gmail.com, *[c]oghenerume.ogolo@nileuniversity.edu.ng, [d]ike.okafor15@gmail.com, [e]dr.abdullahi.gimba@gmail.com, [f]prejohn73@gmail.com

**Keywords:** pH; drilling fluids; Trona, additives; sodium carbonate; monohydrate process

**Abstract.** Drilling additives play a unique role during drilling operations, from aiding in the control of various drilling challenges to successfully enhancing downhole drilling efficiency. pH enhancers are amongst the plethora of additives imported into Nigeria at exorbitant prices to aid in drilling operations. These additives includes NaOH, $Na_2CO_3$, $Ca(OH)_2$ etc. These additives are used to improve the mud pH and mitigates drill string corrosion. The high cost of importation of these additives, has warranted the need for product substitution which should take advantage of the locally available resources. This paper evaluated the suitability of locally-sourced Trona, as a mud additive in drilling mud. Trona is known chemically as Sodium Sesquicarbonate or Sodium Hydrogen Carbonate. A distinguishing factor in this research work was the purification of Trona by extracting the compound of interest ($Na_2CO_3$) from it using the monohydrate process. The purification method involved crushing and screening of Trona as well as calcination, filtration and evaporation processes. The analysis of the Trona and the extracted product was performed using quantitative analysis and characterization tools such as FTIR and EDX. Further experimentation was carried out to evaluate the effects of the extracted sodium carbonate on the mud pH, rheology, and density of the water-based mud. The results were also compared to the results gotten from the addition of conventional $Na_2CO$ to similar mud samples. The extracted $Na_2CO$ was observed to increase the pH of the mud samples from 8.73 to 9.52 and the commercial $Na_2CO$ increased it from 8.73 to 10 and this value is still in the range of API standard. The pH enhancers from both sources also had effect on the mud rheological properties. This indeed showed that the extracted $Na_2CO$ from Trona acted as drilling fluid pH enhancer and hence possess the potential for usage in the industry.

## 1.0 Introduction

Drilling fluid as a terminology, encompasses all of the compositions of fluids used to the removal of cuttings from the wellbore [1]. Since the time of discovery of petroleum, different techniques have been developed in the industry over the years, to bring about safe and successful drilling operations. Drilling fluid is part of such technologies [2]. What started out as a mixture of water and clay in the early era of drilling operations, has evolved into a complex mixture involving the use of organic and inorganic products [3,4]. These complex drilling fluids play several functions simultaneously [5,6,7]. They perform numerous function during drilling operations. There are used to clean the wellbore, carry and suspend cuttings, prevent caving, lubricate the bit, ensure tightness of the well wall and form an impermeable cake near the well bore [4-16]. To perform these functions effectively, the properties of the mud have to be continuously monitored and modified to suit the drilling conditions. These modifications are done using additives [16,17].

pH enhancers are additives used to mitigate the problems of corrosion during drilling especially when drilling through formations with acid gases. pH is an articulation of the alkalinity and acidity of a solution measured between 0 and 14 with 7 being neutral [3]. Having the right mud pH will help to prevent corrosion of the drill bits [3]. Standards additives used for pH enhancement includes: Sodium carbonate, sodium hydroxide calcium hydroxide etc. A well-known source of sodium carbonate is Trona mineral. Trona is a naturally occurring mineral, known chemically as sodium sesquicarbonate ($Na_2CO_3.NaHCO_3.2H_2O$) or sodium hydrogen carbonate ($Na_3H(CO_3)_2.2H_2O$), it is an evaporative mineral occasionally encountered as a saline lake deposit [18,19]. In Nigeria, Trona can be found in the North-Eastern States i.e. Borno, Yobe, Taraba, Adamawa and Bauchi State [20]. The chemical composition of Trona from Borno state was analyzed and said to contain 20% $Na_2CO_3$ [20].

Okorie (2009) recommended the use of Trona as a drilling mud additive because his experiment yielded positive results. He concluded that the addition of raw Trona to local drilling mud increased the pH from 6.98 to 12.86. He also inferred that the local modifier is environmentally friendly as they are used to prepare food in the domestic setting. As a result, he urged the use of local pH materials as it would lead to the development of Nigeria content drive as well as further applications in the West African Sub-region. The experiment he conducted was performed without prior purification of Trona. As seen from literature, purification of Trona would likely lead to better performance of the additive as a pH enhancer as the purification process will help to prevent the effect of gang minerals present in the raw Trona from affecting other mud properties. The purification process will yield sodium carbonate which is among the commercial pH enhancers currently been used in the industry.

This research work investigated the performance of $Na_2CO_3$ extracted from Trona on the mud properties. Trona will be purified and processed using the monohydrate process in order to extract sodium carbonate, also known as soda ash. This involves calcination and filtration processes [21]. Characterization studies of the processed soda ash will be done using Fourier Transform Infrared Spectroscopy (FTIR) and Energy Dispersive X-ray Spectroscopy (EDX). The values of silica, carbonate, bicarbonate, chlorides and sulfate ions in raw Trona sample will be determined using quantitative analysis. The effect of the processed Trona will be tested on water based mud samples prepared using foreign bentonite clay. Comparative analysis of the effects of the purified Trona to imported $Na_2CO_3$ on the mud properties was be carried out.

## 2.0 Theory

### 2.1 pH Enhancement

pH refers to the indication of the acidity and alkalinity of a solution. It is defined as a measure of the concentration of hydrogen ion in a solution. Hence, the higher the pH value, the lower the hydrogen ion concentration, and vice versa. The standard pH scale is 1 to 14 invented by a Danish biochemist called Soren Sorensen. A solution with pH less than 7 is acidic and a solution with a pH greater than 7 is basic. A solution with a pH of 7 is regarded as neutral with pure water being a prime example. Contrary to popular belief, the pH value can be less than 0 or greater than 14 for very strong acids and bases respectively [22].

$$\mathrm{pH} = -\log [H^+] \quad (1)$$

where:

$H^+$ = molar hydrogen ion concentration

The pH of drilling mud is vital as it helps in the mitigation of corrosion of the drill strings during drilling operations. pH can be measured using a pH paper or a digital pH meter however, a pH meter is recommended because it gives more accurate pH values. Ideally the mud pH should lean towards the alkaline portion of the pH scale, however, the optimum pH for any mud system during drilling operations depends on the type of mud in use and the nature of the formation being drilled [23]. API specification for mud pH is between 9.5 and 12.5 [24]. A decrease in the pH level of the mud is characterized by an increase in the level of acid gases (e.g. $CO_2$, $H_2S$ e.tc.) in the wellbore. These acid gases form acids in water and adversely affect the mud pH causing problems such as thickening, dispersion, flocculation and clay separation [3]. Sodium carbonate increases the pH of the mud sample according to the following equations:

$$Na_2CO_3 + H_2O \Rightarrow Na_2O + H_2CO_3 \quad (2)$$

$$Na_2O + H_2CO_3 \Rightarrow H_2O + CO_2 + NaOH \quad (3)$$

### 2.2 Trona

The deposit of Trona ore is a body of rock that contains metallic compounds or native metals in sufficient quantities that has an economical value. It is an ore in which metals can be profitably extracted from [25].Trona, also called sodiumsesquicarbonate is a solid material found in nature in form of double hydrate salts of $Na_2CO_3$ and $NaHCO_3$ having chemical formula $Na_2CO_3.NaHCO_3.H_2O$[26,27,28,29]. Trona is non-flammable, white, yellow-gray or brown crystalline solid with non-odour, it has solubility of 13g/100ml of water at 0 °C and 42g /100 ml of water at 20 °C. Mineral Trona comprises of primarily of 80-95% sodium seisquecarbonate ($Na_2CO_3.NaHCO_3.2H_2O$) and in lesser amounts of sodium sulfate ($Na_2SO_4$), sodium chloride (NaCl), as well as small amounts of organic matter and insoluble materials such as clay, shales and sand. Sodium Sesquicarbonate ($Na_3H(CO_3)_2$) known as trisodiumhydrogendicarbonate, is a double salt of sodium carbonate and sodium carbonate and has a needle-like structure. Trona which is popularly known as Akanwu or potash in Nigeria, is one of the complex salts which is used for the production of soda ash.

#### 2.2.1 Purification of Trona

Trona can be purified to produce soda ash using the monohydrate, sesquicarbonate, carbonation and alkali extraction processes. However, the most common processes used are the monohydrate and sesquicarbonate processes. Both process undergo the same unit operations but in a different order. Although $Na_2CO_3$ can be produced from limestone and salt, both of which are practically inexhaustible, synthetic $Na_2CO_3$ is costlier to produce and it also generates environmentally deleterious wastes [30].

The monohydrate process involves crushing and screening the Trona ore. The screened ore is then calcined to convert the sodium bicarbonate into sodium carbonate, and dissolved in warm water. The solution is left to settle and the top is decanted and filtered. The filtered solution undergoes evaporative crystallization to precipitate sodium carbonate monohydrate. The monohydrate slurry is centrifuged and the crystals are moved to the bottom of the mixture. The crystals are placed in the drying ovens to produce sodium carbonate. A brief description of the processes involved in the monohydrate process are as follows;

**a. Calcination**

Calcination refers to the thermal decomposition of solids to remove chemically bound water and carbon dioxide or other gases present in the solid materials [30]. Calcination is a very important step in the production of soda ash from relatively cheap Trona ore. It is present in the main methods of Trona purification as it helps to eliminate gangue minerals present in the Trona ore. It involves heating Trona to an appropriate temperature to remove carbon dioxide and water. During the process, weight loss is experienced and this has been attributed to the loss of water and carbon dioxide. The weight loss could reach up to 25.44 % after complete calcination. [31]. Experimental results show that weight loss (%) increases with calcination temperature and decreases with particle size [31]. Production of the probable main product, dense soda ash, from Trona requires the stoichiometry as shown below;

$$2(Na_2CO_3.NaHCO_3.2H_2O) \Rightarrow 3Na_2CO_3 + 5H_2O + CO_2 \quad (4)$$

**b. Decantation and Filtration**

Sedimentation allows insoluble heavier particles in a mixture to settle at the bottom of a container, after being left undisturbed for a period of time. Decantation is the process of separating mixtures that are made of immiscible liquids or a solid and a liquid. It involves obtaining a clear liquid after sedimentation, and transferring it to another container without disturbing the settled layer. Filtration involves the separation of a suspended solid matter from a liquid by passing the liquid through a porous membrane called a filter. It is used to achieve further separation after the decantation process. All separation processes above are physical processes that do not require any chemical reaction. After calcination, the calcined Trona is dissolved in water and undergoes sedimentation, decantation and filtration.

**c. Evaporative Crystallization**

This refers to the transition of the sample from liquid to solid phase as a result of the evaporation of the solvent. The molecules of the liquid sample when subjected to the suitable temperature arrange themselves in highly organized and tightly bonded structures. The formation of a highly ordered structure prevents the incorporation of foreign particles into the lattice, resulting in a solid product of high purity. The principle of crystallization is based on the limited solubility of a compound in a solvent under certain conditions. Evaporative crystallization is the most popular method of crystallization used [32]. This method requires a high concentration of the desired product in the solution. Therefore, the ratio of the solute mass to the solvent mass should be high. After filtration, the sodium carbonate solution undergoes evaporative crystallization which would lead to the production of sodium carbonate monohydrate crystals and accompanying liquor.

**d. Centrifugation**

Centrifugation is a process whereby components in a mixture are separated through spinning. This process uses a device called a centrifuge for the separation. At a fixed liquid viscosity and centrifugal force, the rate of sedimentation of a particle will be proportional to its molecular weight and to the difference between the density of the solution and the particle density. Centrifugation occurs based on the rotor speed, shape, size, and density of the particles and the nature of the medium. During Trona purification, the monohydrate slurry gotten after crystallization is separated using the centrifuge.

## 3.0 Methodology

This section focuses on the different processes carried out to study the potential of sodium carbonate extracted from Trona as a drilling fluid additive. Trona was collected from Yobe State, Nigeria, processed and characterized using FTIR and EDX analysis. The compound of interest, sodium carbonate, was extracted from Trona using the monohydrate process and characterized as well. Several samples of water-based mud were prepared using foreign bentonite clay and distilled water. Varying amount of the extracted and imported sodium carbonate (1.5 g - 3.5 g) were added to each of the mud samples respectively to determine the effect of addition of both the extracted and imported sodium carbonate on the mud properties.

### 3.1 Materials and Equipment

The equipment used for the experiment of this research work are listed in Table 1.

**Table 1:** Equipment used for the experiment

| Equipment | Uses |
|---|---|
| U-Test Compaction mould and rammer | Crushing samples |
| 0.704 mm and 0.106mm UTest Laboratory test sieve assembly | Sieving clay samples |
| Laboratory Mud mixer (Hamilton Beach Commercial) | Mixing mud samples |
| Weighing machine | Measuring weight |
| Titration stand | Titration. |
| Cole Parmer Box Furnace (Model CBFM516C) | Calcination |
| Hot plate | Heating |
| Hermle Centrifuge (Model Z 300) | Centrifugation |
| Drying oven (U-Test GENO DT104A) | Drying |
| OFITE Atmospheric Mud balance | Measuring mud density |
| OFITE Testing equipment Inc. (Model 900 Viscometer) | Measuring rheological properties |
| Digital pH meter (Mettler Toledo LE438) | Measuring mud pH |
| Fourier Transform Infrared Spectroscope, ThermoFisher Scientific (Model Nicholet iS5) | Determination of functional groups |
| OFITE Sample Compacter | Compressing sample into pellets |
| Vacuum filtration pump | Filtration |

### 3.2 Trona Sample Preparation and Characterization

#### 3.2.1 Preparation of Trona

Trona was mined along Jajare stream, Panami village, Gashuwa LGA, Yobe State, Nigeria and taken to the lab for preparation. Samples of Trona were crushed and sieved to remove particles like dirt, pebbles and stones and analysed in the laboratory.

#### 3.2.2 Quantitative Analysis of Trona

Constituents of Trona was determined according to the standard methods of quantitative analysis. Standard titrants and indicators were used in this analysis. The content of sodium carbonate and sodium hydrogen carbonate were determined volumetrically by titrating against hydrochloric acid (HCl). The content of sodium sulfate and sodium chloride were determined volumetrically using barium chloride (BaCl) and silver nitrate ($AgNO_3$) solutions respectively. Mass of insoluble solids were determined gravimetrically by dissolution, filtration and drying.

#### 3.3.3 Extraction of Sodium Carbonate from Raw Trona Sample

Steps to extract sodium carbonate from raw Trona was adapted from processes postulated by Madima (2009) and Örgül (2003). About 306 grams of prepared Trona sample was measured into a ceramic crucible using a measuring balance and calcined in a furnace for 90 minutes at 400°C. After calcination, the sample was allowed to cool. The sample was then dissolved in distilled water ensuring the solution becomes saturated. The now saturated solution was left for 24 hours to ensure complete dissolution of the sodium carbonate compounds present. The solution was observed to be separated with insoluble solids at the bottom and a clear yellowish solution at the top. The top solution was decanted into a beaker and filtered using a vacuum filtration pump. The decanted solution was poured gently into the filtration funnel, depositing yellowish clear solution into the filtration bottle. The clear solution was transferred into a beaker and placed on a hot plate for evaporative crystallization. The solution was heated at 100 °C until crystals were formed. This mixture was placed in a centrifuge and spun at 4500 rpm for 10 minutes. After centrifugation, the top solution was decanted and the bottoms (crystals) was heated at 150 °C for 30 minutes in a drying oven. Evaporative crystallization was performed on the decanted top solution, again to produce more crystals. The final product was allowed to cool, and weighed.

#### 3.3.4 Characterization Studies: Fourier Transform Infrared Spectroscopy (FTIR) Analysis and Energy Dispersive X-ray Analysis

FTIR is characterization tool that is used for the identification of organic and inorganic chemicals. It is one of the most common spectroscopic techniques that identifies the chemical functional groups and can be applied to either fluid or solid samples. The process of characterizing involves producing a beam of light, splitting it and passing one through a reference and the other through the sample of interest. The beams are reflected back towards a detector, signals are compared and results are obtained. Compounds are then identified by matching a reference spectrum with the spectrum of the unknown sample. This characterization method is based on the principle that molecular bonds vibrate at different frequencies depending on the elements and bond type [21]. Characterization studies using FTIR done for the raw Trona sample, calcined Trona sample and the extracted sodium carbonate. For FTIR analysis, the samples were prepared by first of all mixing the samples with potassium bromide (KBr) and compressing to form pellets. These pellets were placed in the spectroscope and analysed.

EDX is a characterization tool that helps to identify the elemental composition of a sample which can be expressed quantitatively as a percentage of the total identifiable composition of the sample. Analysis is done by bombarding the sample with an electron beam within the Scanning Electron Microscope. This collision would lead to the displacement of some of the electrons which triggers a replacement by an electron from a higher energy level. This replacement leads to a loss of energy that is referred to as an x-ray. The amount of energy of the x-ray is unique for the atom of every element. The output of an EDX analysis is the EDX spectrum, and it displays peaks corresponding to the energy levels received. Each peak is unique to an atom and corresponds to a particular element. Characterization studies using EDX was carried out for the raw Trona sample, calcined Trona sample and the extracted sodium carbonate.

### 3.4 pH Test of Raw and Calcined Trona, Extracted and Commercial Sodium Carbonate

To determine the efficacy of the extracted sodium carbonate, 1wt% solution of raw Trona, calcined Trona, extracted product and conventional sodium carbonate was prepared and the pH of each solution was tested using a digital pH meter.

### 3.5 Mud sample preparation

Mud samples were prepared by mixing 350 mls of water with 17.5 g of foreign bentonite clay. The beneficiation process was done using the experimental set up highlighted in this section.

- Mud sample containing no additive
- Mud sample + 1.5 g of extracted sodium carbonate
- Mud sample + 2.5 g of sodium carbonate
- Mud sample + 3.5 g of sodium carbonate
- Mud sample + 1.5 g of conventional sodium carbonate
- Mud sample + 2.5 g of conventional sodium carbonate
- Mud sample + 3.5 g of conventional sodium carbonate

### 3.6 Measurement of mud properties

To evaluate the performance of the extracted and conventional sodium carbonate on the mud properties during the mud beneficiation process, properties of the mud were tested for according to API Recommended Practice 13B-1 (2009) after beneficiating the mud using the experimental design developed for the experiments. The properties of the mud tested include the mud pH, mud density, and mud rheology [33].

## 4.0 Discussion of Results

### 4.1 Analysis of Trona

Volumetric analysis was performed to determine the content of sodium carbonate, sodium bi-carbonate, sodium chloride and sodium sulphate compounds as well as the total alkalinity of the Trona sample. The Trona solution analysed was allowed to dissolve for 24 hours to ensure that there is complete dissolution of the Trona in the water.

**Table 2**: Volumetric analysis of Trona

| Test | Volume of Titrant (mL) | | | Colour change |
|---|---|---|---|---|
| | 1st Run | 2nd Run | Average value | |
| Total Alkalinity | 7.50 | 6.60 | 7.05 | Red to blue-black |
| Sodium bicarbonate | 48.20 | 48.40 | 48.30 | Pink to transparent |
| Sodium Chloride | 2.60 | 2.60 | 2.60 | Pink to Cloudy-white |
| Sodium Sulphate | 3.40 | 3.60 | 3.50 | Pink to Cloudy-white |

Table 2 shows the volumetric analysis of the Trona samples Total alkalinity refers to the content of alkaline compounds in a substance. To determine the total alkalinity, Trona solution was titrated against HCl solution. HCl solution reacts with all the sodium bicarbonate present in the Trona sample. It is assumed that majority of the alkaline nature of the Trona solution is indicated by sodium bi-carbonate compound present. The volume of the titrant is used to calculate the concentration of alkaline compounds present in the solution using Avogadro's law.

In determining the sodium bicarbonate content, Trona solution is titrated against excess NaOH to convert all the $HCO^{3-}$ ions to $CO^{3-}$ ions. The $CO^{3-}$ ions were then selectively precipitated by reacting it with $BaCl_2$. The remaining solution containing NaOH, NaCl and $H_2O$ was then titrated against HCl to obtain the volume of the excess NaOH. Using the volume of the excess NaOH, the concentration of the sodium bicarbonate present in the solution was calculated. Total alkalinity is the sum of the concentrations of sodium carbonate and sodium bicarbonate in the sample, therefore the concentration of sodium carbonate present in the Trona solution can be determined. The volumes of sodium chloride and sodium sulphate in the sample were calculated using the volumes of their titrants respectively, with the Avogadro's law.

Using titrimetric calculations, the mass of the compounds analysed were calculated. The following calculations were used;

$$5.0\ C_A V_A = C_B V_B \quad (5)$$

$$6.0\ n = CV \quad (6)$$

$$7.0\ n = \frac{M}{M_m} \quad (7)$$

where:
n = number of moles
C = Concentration (M)
V = volume (L)
M = Mass (g)
$C_A$= Concentration of Trona solution (mol/L)
$C_B$= Concentration of Titrant (mol/L)
Mm = Molar mass (g/mole)

**Table 3**: Chemical composition of Trona

| **Compounds** | **Mass(g)** |
|---|---|
| $NaHCO_3$ | 0.5712 |
| $Na_2CO_3$ | 1.7382 |
| NaCl | 0.6076 |
| $Na_2SO_4$ | 1.9880 |
| Sand and other Insoluble solids | 17.6500 |
| **TOTAL** | 22.5550 |

Table 3 shows the chemical composition of the Trona sample analysed content. This is as a result of the quality of the sample analysed. Trona sample mined from the surface has greater sand content that those mined from greater depths. From the data in Table 3, the mass occupied by the sodium element was calculated to be 1.7937 g which translates to 7.9367% of the total sample analysed. Mass occupied by the carbon content was also calculated to be 1.234 g that is 1.23% of the sample analysed 22.6 g of Trona sample was analysed but only 22.555 g was accounted for. This is due to spillages during analysis.

## 4.2 Extraction of Sodium Carbonate from Trona Sample

**Table 4**: Mass change during extraction

| | |
|---|---|
| Mass of raw Trona sample (g) | 306.04 |
| Mass of Calcined Trona sample (g) | 278.15 |
| Mass loss after calcination (g) | 27.89 |
| Weight loss after calcination (%) | 9.11 |
| Mass of extracted sodium carbonate (g) | 19.6 |

Table 4 shows the mass change during the extraction process. About 306 grams of Trona sample was calcined in a furnace at 400 °C [31]. The calcination process was to increase the sodium carbonate content in the sample as addition of heat leads to decomposition of the sodium bicarbonate into sodium carbonate. After calcination, 27.89 grams of the sample was lost due to removal of by-products; $H_2O$ and $CO_2$. This resulted to 9.11% weight loss after calcination explained by removal of crystal water and exposure of $CO_2$ according to equation 4.

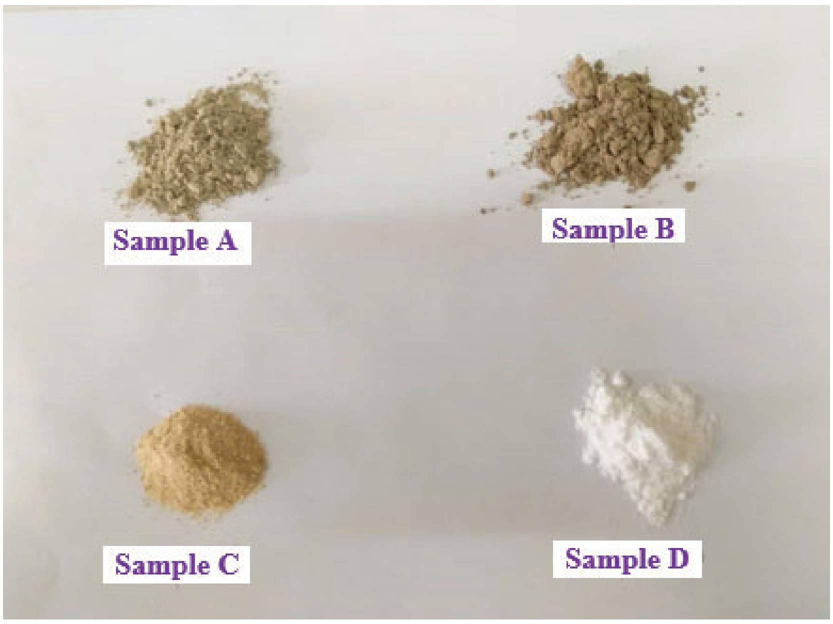


**Figure 1**: Raw Trona (Sample A), Calcined Trona (Sample B), Extracted Soda ash (Sample C), Imported Soda ash (Sample D)

Figure 1 shows samples of raw and calcined Trona sample along with the extracted and imported soda ash (what sample corresponds to what picture? E.g. Sample A is raw Trona). It was observed that the raw sample is light brown in colour. Upon calcination, the sample became dark brown. The calcined sample was dissolved in 350 ml of water to dissolve the soda ash and allow the insoluble materials to be separated from the soda ash solution [30]. The resulting solution was dark yellow and after filtration, insoluble solids were removed and the filtrate attained a light yellow colour. The filtrate was heated on a hot plate at 100 °C to precipitate the sodium carbonate monohydrate crystals [21]. Other dissolved impurities, such as sodium chloride or sodium sulphate, remained in solution so the solution was heated and centrifuged again to precipitate more crystals. The crystals and liquor were separated and the sodium carbonate monohydrate crystals were heated in a drying oven to remove water of crystallization. 19.6 g of sodium carbonate was extracted from the Trona sample calcined which is translated to a yield of 6.4%.

### 4.4 pH Test of Raw and Calcined Trona, Extracted and Commercial Sodium Carbonate

pH tests were performed on the samples listed in the Table 5. 1 weight % solution of the samples were prepared and used for this analysis.

**Table 5**: pH values

| **Samples tested** | **pH** |
|---|---|
| Raw Trona | 9.38 |
| Calcined Trona | 9.60 |
| Extracted sodium carbonate | 9.93 |
| Imported sodium carbonate | 10.28 |

From the Table 5, it is seen that there is an increase in the pH value of the samples tested. After calcination, the pH value increased by 2.3%, indicating an increase in the alkaline content. Filtration processes increased the pH value by 5.2% and the pH at this point was 9.93 indicating the removal of some impurities in the final product. The pH value of the imported soda ash was 10.28. This difference in pH values between the extracted soda ash solution and the imported soda ash solution could attributed to the presence of some impurities still present in the extracted soda ash or due to concentration difference as minerals composition and concentration vary with location and depth.


**Figure 2**: 1 wt % solution of the samples used for the pH test

From Figure 2, it is seen that the solution containing imported sodium carbonate is clear. There was complete dissolution of the imported pH enhancer in water. The same can be said for the extracted sodium carbonate solution tested however, the solution maintained a yellowish color as a result of some impurities still present in the solute.

## 4.4 Characterization Studies

### 4.4.1 Energy Dispersive X-ray (EDX) Analysis

This characterization tool was used to identify the various elements present in the raw Trona sample, calcined Trona sample and the sodium carbonate extracted. This can be seen in Fig 3-Fig 5. Fig. 3 shows a high level of silica in the raw Trona sample which is attributed to the presence of sand in the sample. Upon calcination, trace elements like molybdenum and iron were reduced. This decrease was accompanied by a slight increase in sodium and oxygen levels as seen in Fig. 4. Fig. 5 shows the effect of the monohydrate process of the Trona sample, the sodium and carbon content increased significantly. The silica content was also reduced. This analysis was performed three times on each samples to acquire average values and repeatability and the average values obtained for each of the samples analysed was shown in Fig 6.

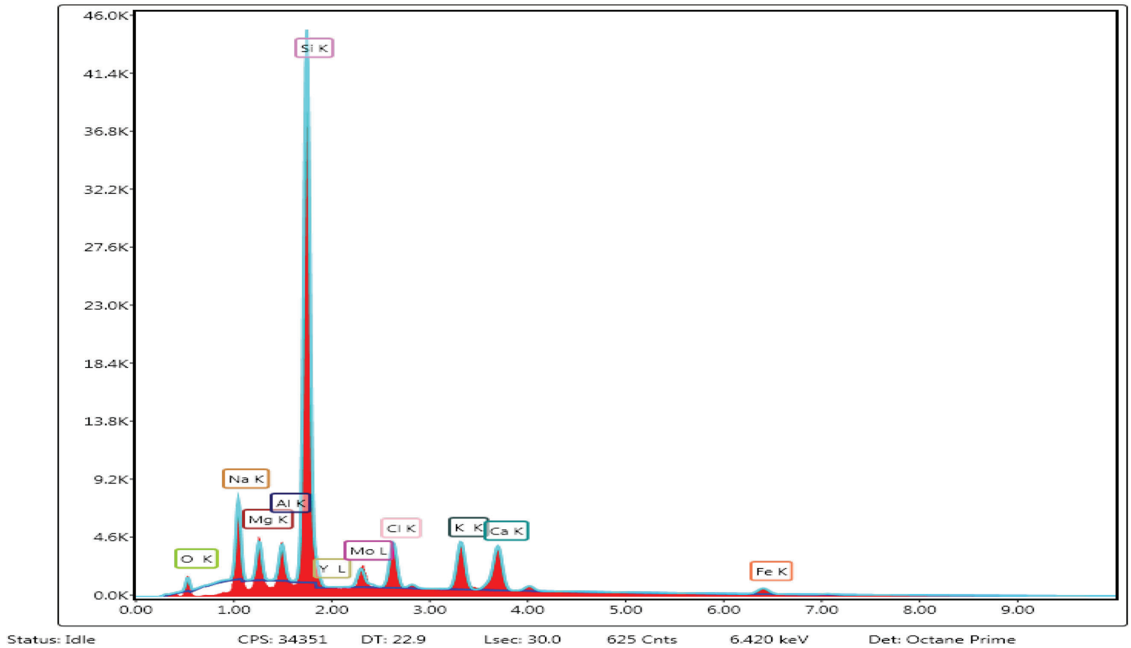


**Figure 3**: EDX spectrum of raw Trona sample (3)

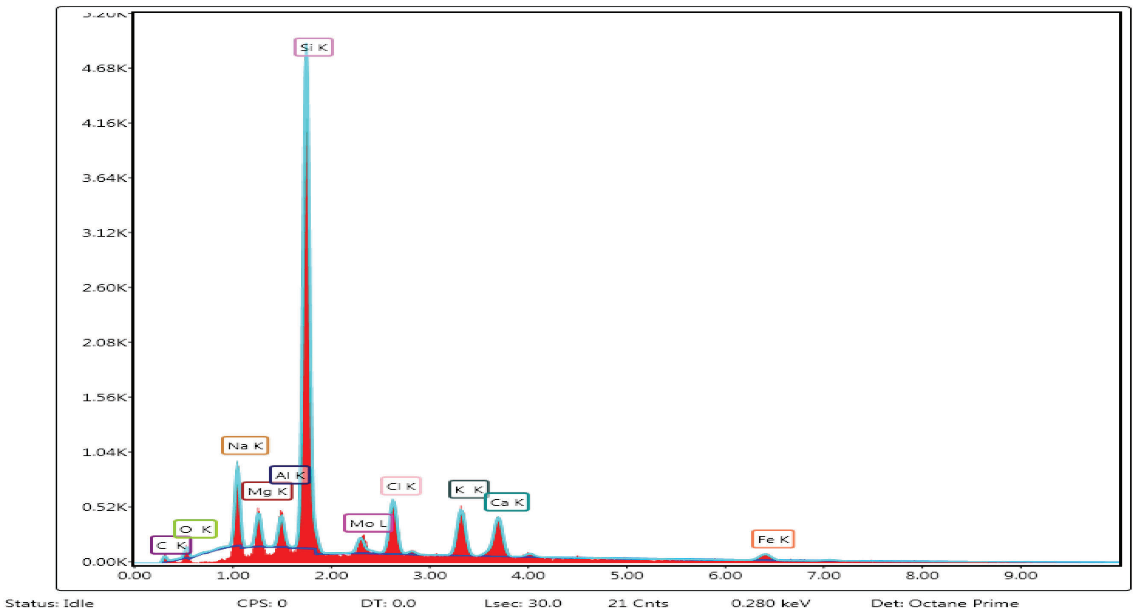


**Figure 4**: EDX spectrum of calcined Trona sample

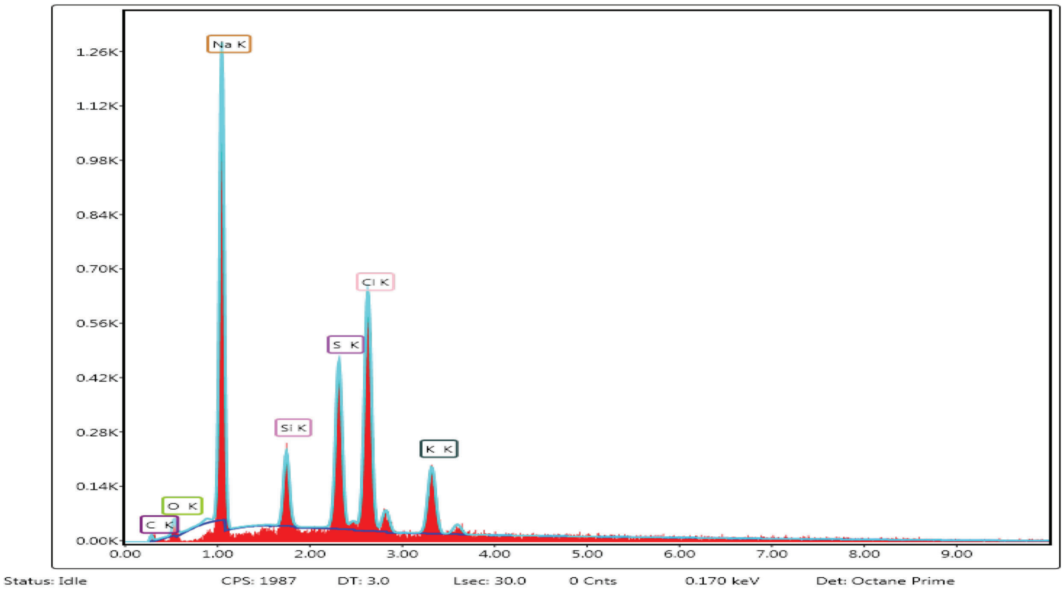


**Figure 5**: EDX spectrum of extracted Sodium carbonate

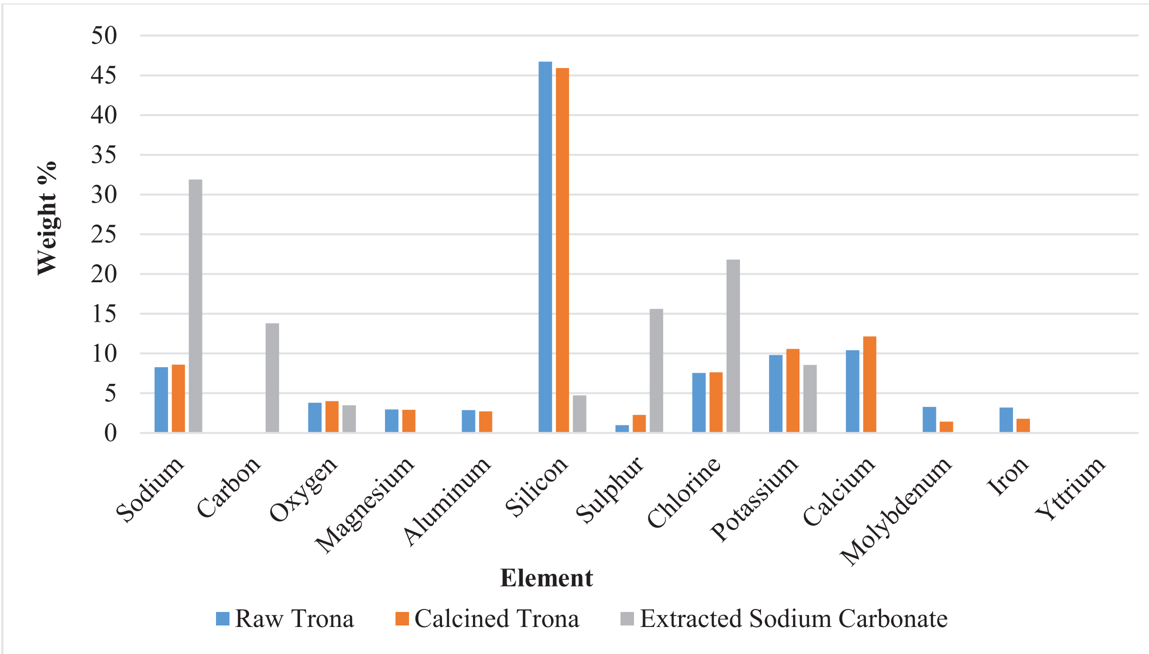


**Figure 6**: Comparison between elements present in raw Trona, calcined Trona and extracted sodium carbonate

Figure 6 contains the average values of the weight % of elements contained in the three samples analyzed. EDX analysis of the Trona samples at different stages showed an increase in the sodium and carbon content as well as a decrease in the silicon content of the sample. After calcination, a 3.99 % increase was observed in the sodium content of the sample analyzed. Carbon content remained unchanged and the oxygen content increased by 5.51 %. Content of Magnesium, Aluminum, Molybdenum, Iron also decreased. while the content of sulphur, chlorine, potassium, and calcium increased after calcination. Yttrium was completely removed. Filtration processes performed eliminated magnesium, aluminum, calcium, molybdenum, iron elements that were present in the calcined Trona. The sodium content increased by 23.62 % after filtration as a result of evaporative crystallization. This process precipitated sodium carbonate monohydrate crystals which were dehydrated to form sodium carbonate. Carbon content increased by 13.68 % while the silicon content decreased by 41.19 %. This is because water was added to the calcined Trona causing the sodium carbonate compound present to dissolve completely. After sedimentation, the top solution containing sodium carbonate was decanted and the insoluble solids settled at the bottom were discarded. These solids are predominantly sand which contains silicon.

**4.4.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis**

This characterization tool was used to identify the organic and inorganic compounds present in the raw Trona sample, calcined Trona sample and the sodium carbonate extracted.

Table 6 and Figure A1 shows the functional groups present and range of peaks observed in raw Trona sample when subjected to FTIR analysis. Functional groups present in the sample include inorganic nitrates and aliphatic hydrocarbons. The following peaks (values shown in Tables 6 and 7) were observed and attributed to their respective bonds according to the IR spectrum chart.

**Table 6**: Absorption bands present in raw Trona

| Absorption band ($cm^{-1}$) | Wave number ($cm^{-1}$) | Types of bond | Comments |
|---|---|---|---|
| 3200 - 3550 | 3462.82 | O – H stretching | Alcohol |
| 1380 - 1390 | 1384.59 | C – H bending | Alkane methyl |
| 1087 - 1124 | 1092.84 | C = O stretching | Secondary alcohol |
| 1030 - 1070 | 1031.53 | S = O stretching | Sulphur oxide |
| 880 ± 20 | 873.63 | C – H bending | Alcohol |
| 850 - 550 | 786.40 | C – Cl stretching | Possible Impurities |
| 1650 - 1580 | 1636.48 | N – H bending | Amine |

**Table 7**: Absorption bands present in calcined Trona

| Wave number ($cm^{-1}$) | Types of bond | Comments |
|---|---|---|
| 3447.56 | O – H stretching | Alcohol |
| 1653.79 | N – H bending | Amine |
| 1078.05 | C = O stretching | Secondary alcohol |
| 1031.53 | S = O stretching | Sulphur oxide |
| 874.10 | C – H bending | Alcohol |
| 784.40 | C – Cl stretching | Possible Impurities |

**Table 8**: Absorption bands in extracted sodium carbonate

| Wave number ($cm^{-1}$) | Types of bond | Comments |
|---|---|---|
| 3462.50 | O – H stretching | Alcohol |
| 1457.77 | C = O stretching | Carbonate |
| 1653.80 | N – H bending | Amine |
| 1092.84 | C = O stretching | Secondary alcohol |
| 877.04 | C – H bending | Alcohol |

Table 7 shows the types of bond found in the calcined Trona. A reduction in the absorbance of the functional groups observed in the uncalcined Trona (Figure A2) was also observed showing the efficacy of the calcination process. Figure A3 shows the IR spectrum of the extracted sodium carbonate and the bond type found in it is shown in Table 8. From Figure A3, the peak with wavelength at approximately 1457.77 $cm^{-1}$ shows the presence of $CO_3^{2-}$ ion [21]. Peak between absorption band 3200 – 3500 $cm^{-1}$ indicates the presence of O-H stretching bonds which may be due to moisture still present in the sample [21].

**4.5 Effect of Extracted $Na_2CO_3$ and Imported $Na_2Co_3$ on the Mud Properties.**

Prepared mud samples were analysed to determine mud properties; mud pH, mud weight and rheology.

### 4.5.1 Mud pH

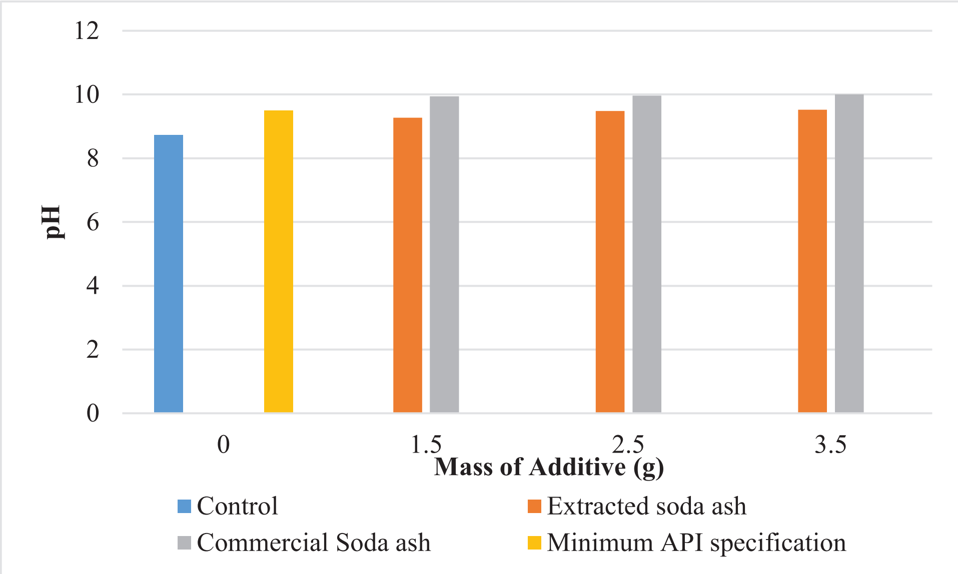


**Figure 7**: pH of Mud samples

As evident in Fig 7, the mud samples prepared with foreign bentonite clay showed an increase in mud pH of 6.19 %, 8.59 % and 9.05 % with the addition of 1.5 g, 2.5 g and 3.5 g of extracted sodium carbonate respectively. This indicates that the extracted additive had impact on the mud pH as expected. 3.5 g of the extracted sodium carbonate increased the pH of the mud sample to meet minimum API standard. The imported pH enhancers increased the pH to meet API specifications as well.

### 4.5.2 Mud density

As seen in Figure 8, the density of the mud remained unchanged despite the addition of varying concentrations of either extracted or imported additives to soda ash. Minimum API specification of mud density (8.65 lb/gal) was not met by any mud samples beneficiated.

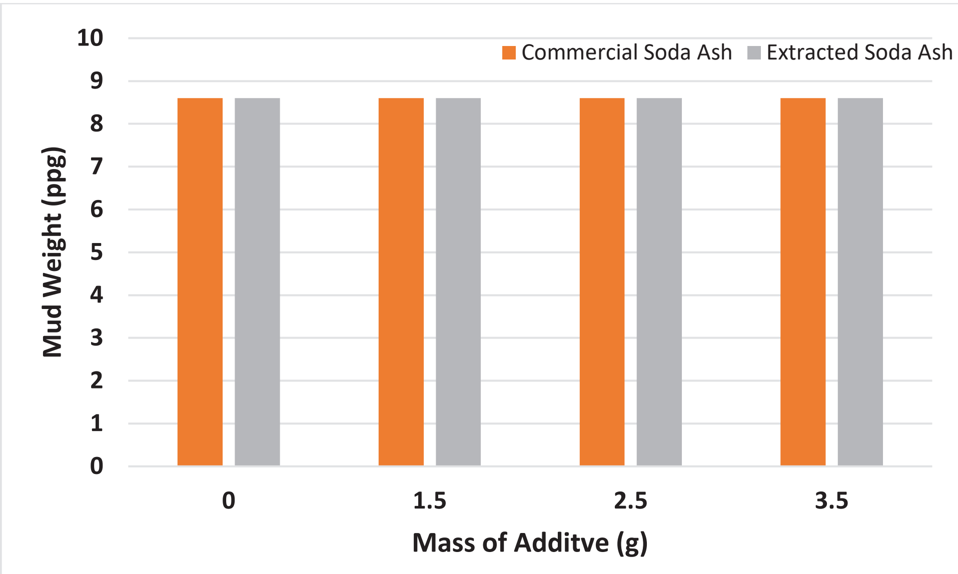


**Figure 8**: Density of Mud samples

### 4.6 Rheological properties

Figures 9 - 11 show the shear stress s. shear rate behaviour of the mud as the additives were added to the mud as varying masses. Figure 9 compares the effect of 1.5 g extracted and imported sodium carbonate on mud sample prepared with foreign bentonite. The extracted sodium carbonate did not cause significant changes to the shear stress behavior, however, addition of the imported sodium carbonate improved the shear stress behaviour of the mud. From Figure 10, addition of imported sodium carbonate (2.5 g) caused a decrease in the shear stress of the mud sample.

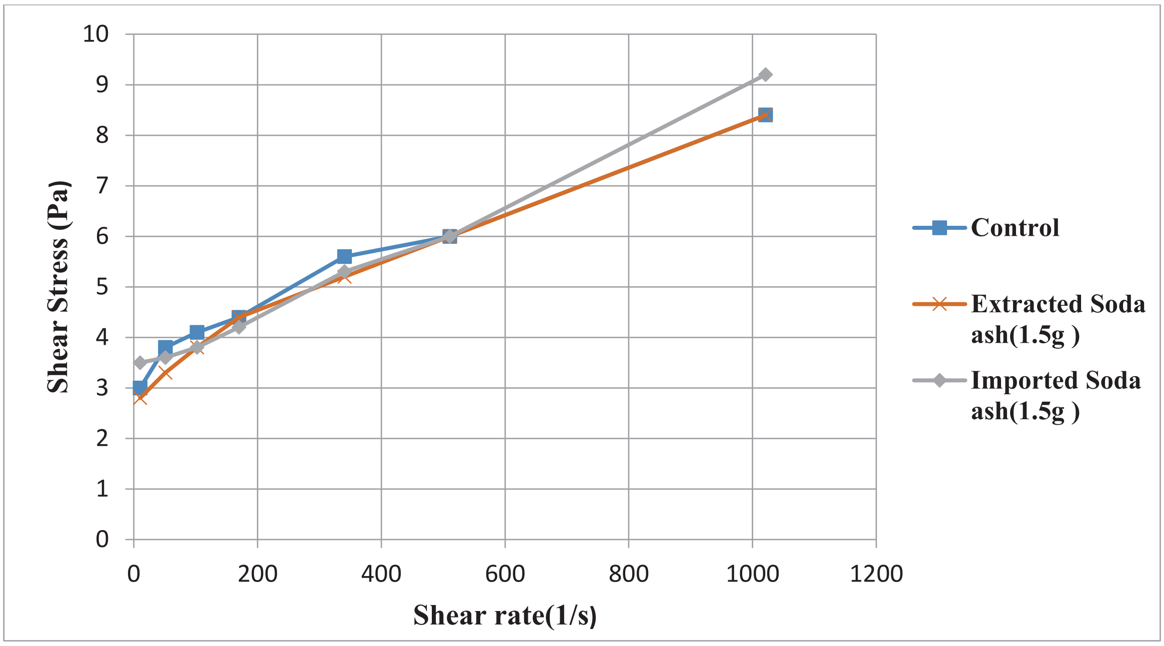


**Figure 9**: Plot of shear stress vs. shear rate for Mud samples containing 1.5 g of extracted and imported sodium carbonate

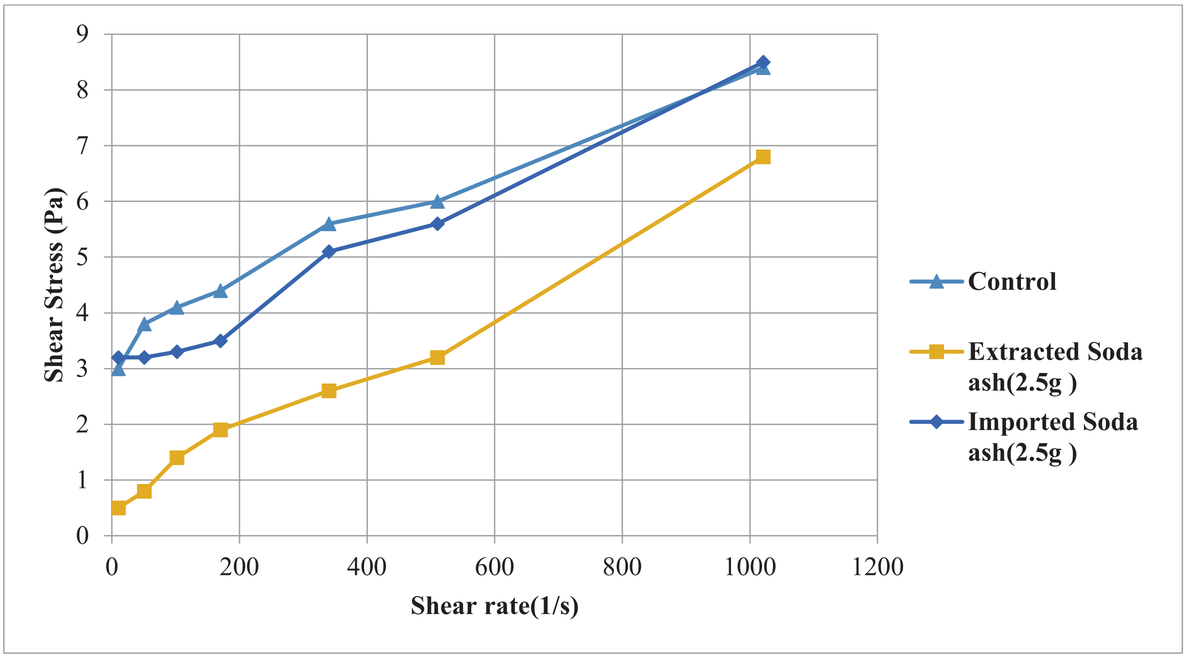


**Figure 10**: Plot of shear stress vs. shear rate of mud samples with 2.5 g of extracted and imported sodium carbonate

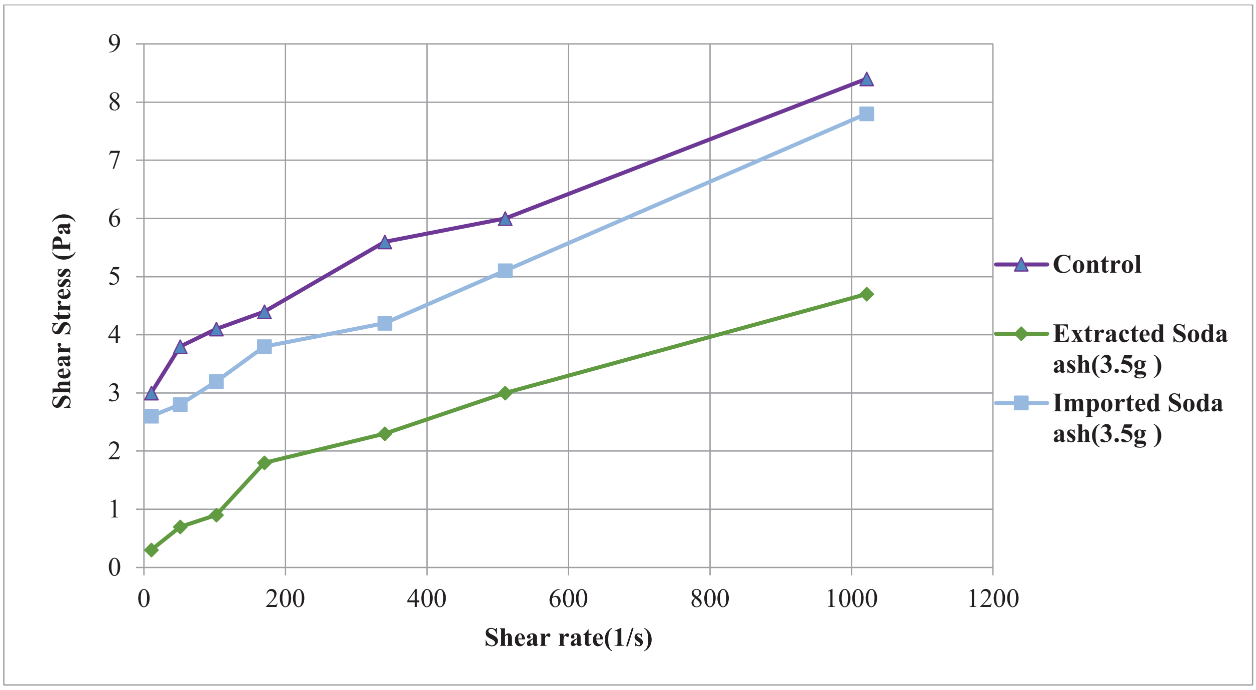


**Figure 11:** Plot of shear stress vs. shear rate for Mud samples containing 3.5 g of extracted and imported sodium carbonate

From Figure 11, addition of imported sodium carbonate (3.5 g) caused a decrease in the shear stress behaviour of the mud samples with the extracted sodium carbonate exhibiting a higher decrease in the shear stress behavior of the mud. From results of the shear stress vs. shear rate, it is deduced that addition of extracted and imported sodium carbonate caused a decrease in the shear stress of the mud samples analyzed.

Figure 12 indicates a decrease in drilling fluid viscosity accompanied by an increase in shear rate. This is a property of a shear thinning fluid. Addition of the imported additive showed an initial decrease in the viscosity which was followed by subsequent decrease following an increase in the concentration of the additive. Addition of the extracted sodium carbonate showed a continuous decrease in the viscosity of the drilling fluid, with increase in concentration.

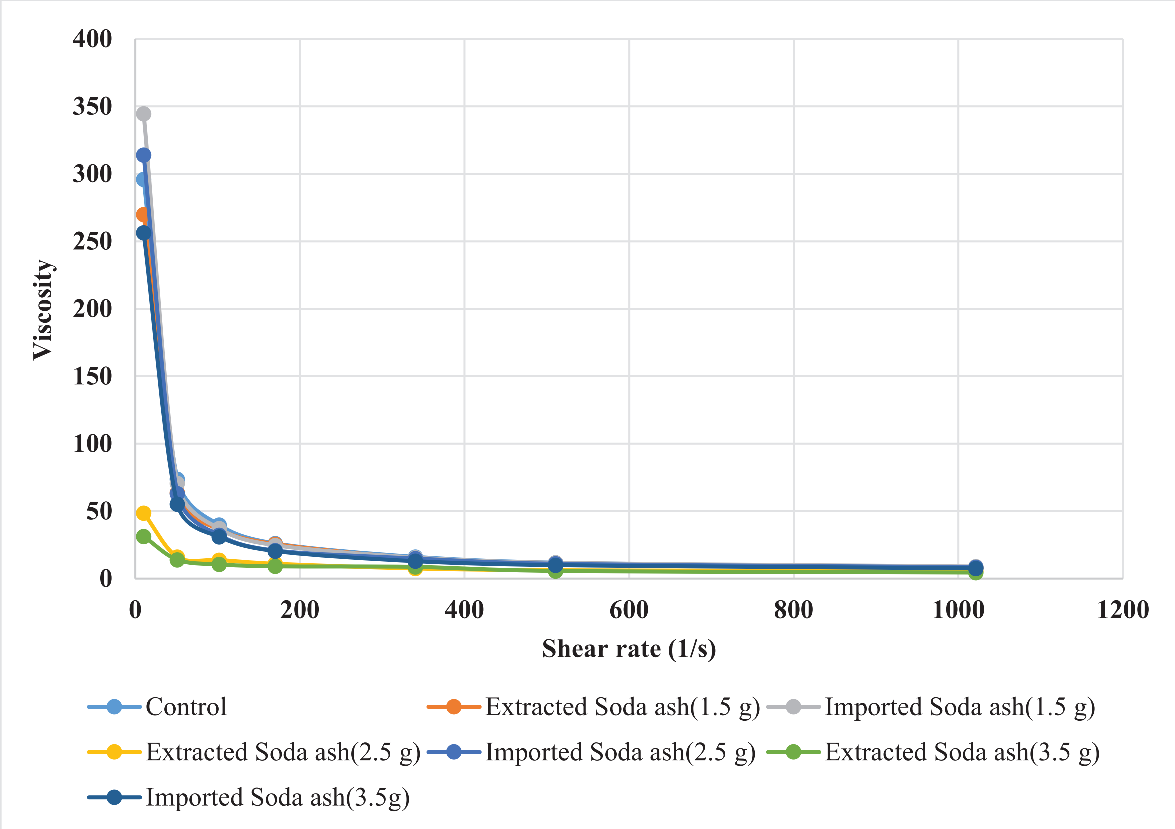


**Figure 12**: Plot of viscosity vs. shear rate of mud samples

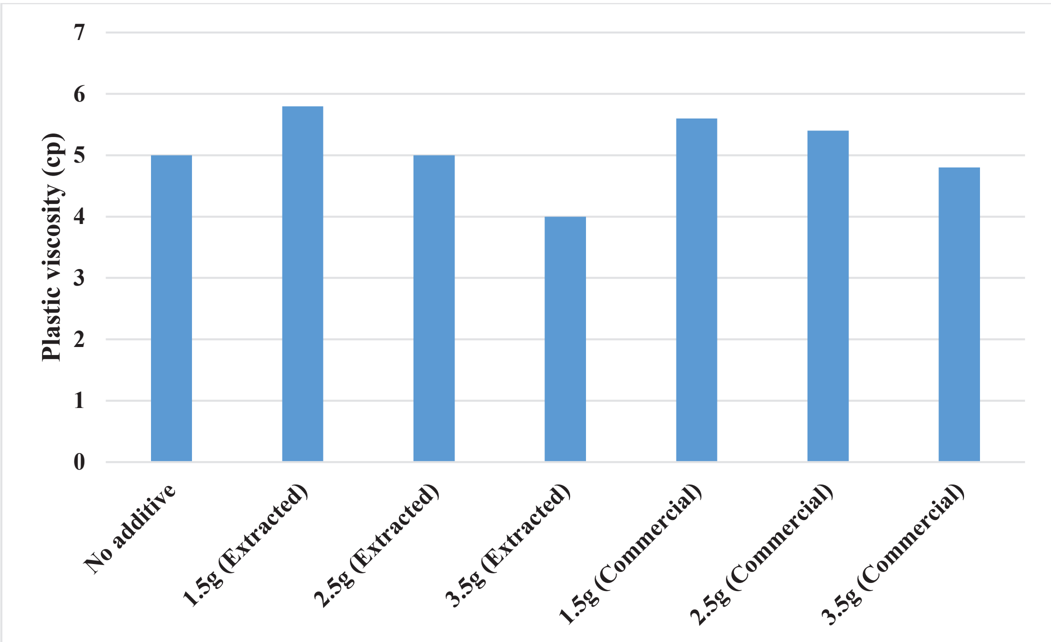


**Figure 13**: Plastic viscosities of Mud samples

From Figure 13, it can be deduced that plastic viscosity of the mud samples decreased with increasing concentration of either extracted or imported sodium carbonate. 2.5 g and 3.5 g of the imported sodium carbonate had greater impact than extracted sodium carbonate of the same concentrations.

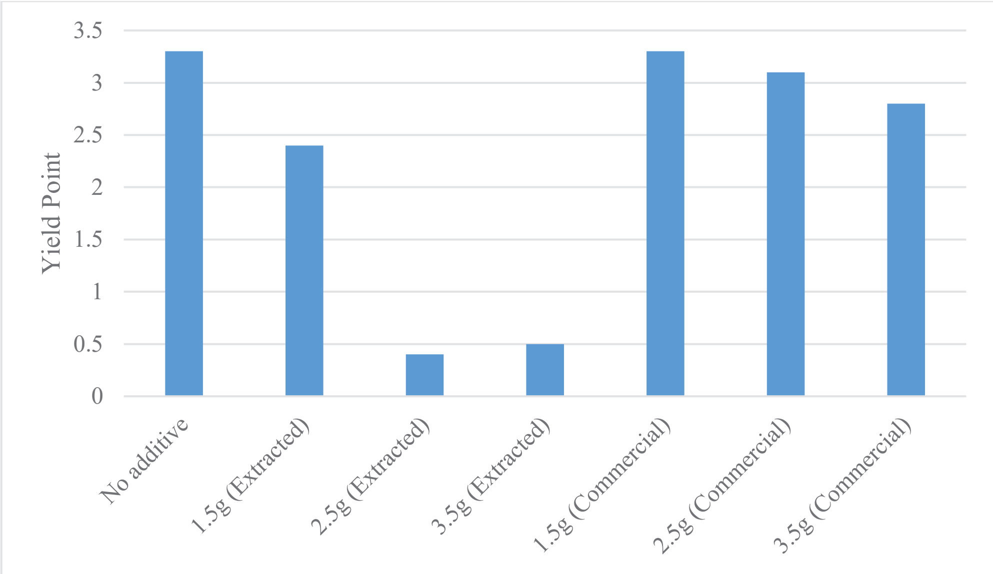


**Figure 14**: Yield points of mud samples

Figure 14 shows that addition of 2.5 g and 3.5 g of the extracted sodium carbonate resulted to a decrease in the yield point of the mud sample. All concentrations of the imported sodium carbonate added to the mud sample caused a decrease in the yield point.

## 5.0 Conclusion

This research work investigated the suitability of locally sourced Trona as a drilling fluid additive. The following were the main concluded points:

- Sodium carbonate was successfully extracted from Nigerian Trona using monohydrate process. The monohydrate process resulted to a yield of 19.6 g which is 6.4% of the total Trona purified.
- The extracted product was analyzed using EDX which showed a 23.29% increase in the sodium content. Carbon content increased by 13.77% and the silicon content reduced by 41.19 %.
- The extracted soda ash from Trona increased the mud pH from 8.73 to 9.52 which is in the range of API standard. The imported sodium carbonate also raised the mud pH to API standard.
- Mud density remained relatively unchanged (8.6 ppg) for all mud samples after the addition of either extracted or imported sodium carbonate.
- Addition of the extracted and imported sodium carbonate had effect on the mud rheology as a decrease in rheological properties was observed upon the addition of both additives.

**Appendix**

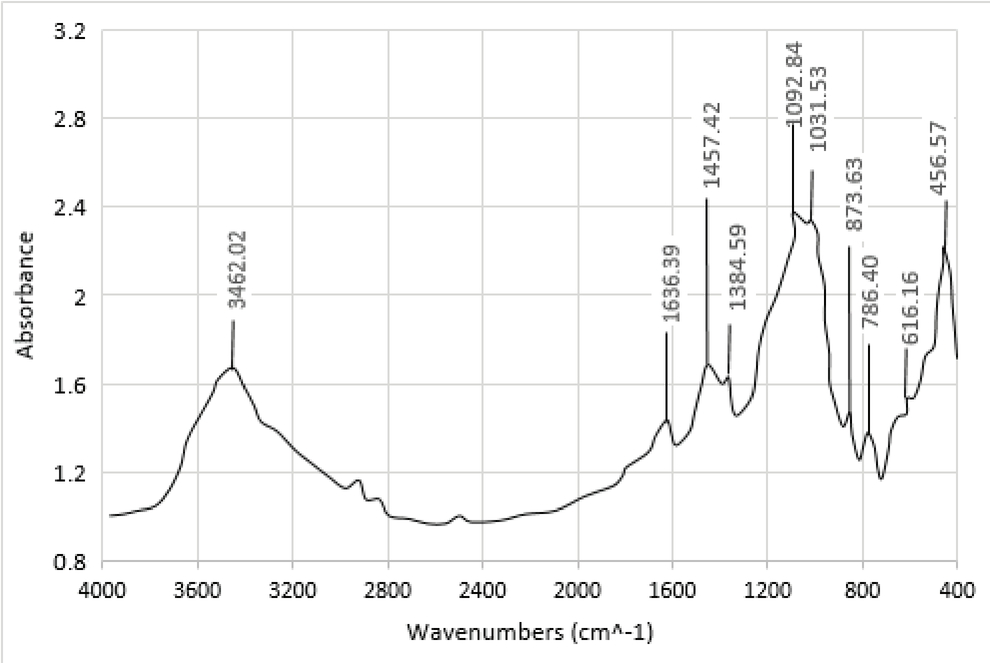


**Figure A1**: Infrared Spectrum of Raw Trona

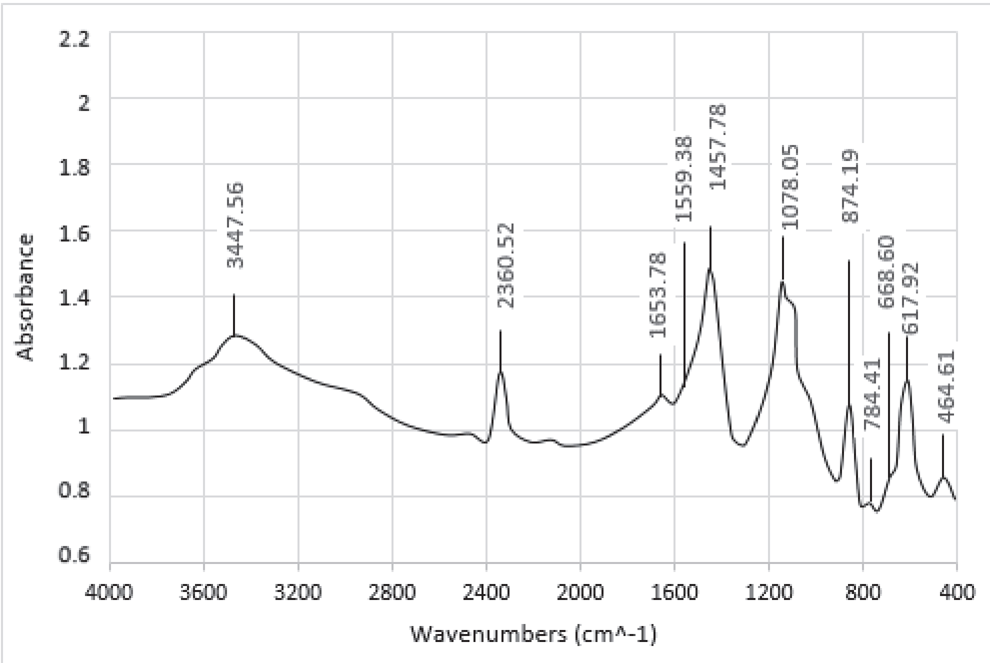


**Figure A2**: Infrared Spectrum of Calcined Trona

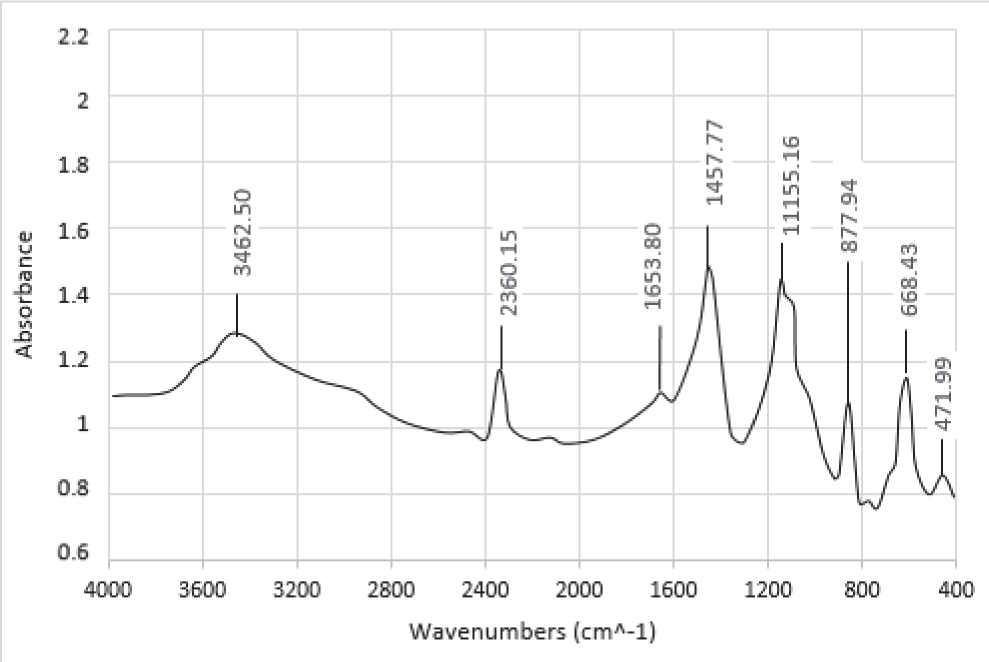


**Figure A3**: Infrared Spectrum of Extracted Sodium Carbonate

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