properties were measured by volume and count using particle size analyzer (Beckmann Coulter L5 200). SEM images of the raw and carbonised coconut shell waste at the various temperatures are indicated in figure 1.

**2.5 X-Ray Diffraction Measurement**

X-ray diffractograms were collected using a sample holder mounted on a Shimadzu Diffractometer (XRD 6000), with monochromatic CuK$\alpha$ radiation ($\lambda$ = 0.1542nm), the generator operating at 40Kv and 30mA. Intensities were measured in the range of $2 < 2\theta < 60^0$, typically with scan steps of $0.3^0$ and $6^0 min^{-1}$. Peak separations were carried out using Gaussian deconvolution. After deconvolution it was possible to calculate and compare several parameters. The d-spacing were calculated using Bragg's equation. The average size of crystallites, Bhkl was estimated from the widths of reflection hkl, using the well-known formula of Scherrer (Popescu et al, 2011). See diffractograms of raw and carbonised coconut shell waste powder at $500^0$C in figure 2 and percentage crystalline index in table1.

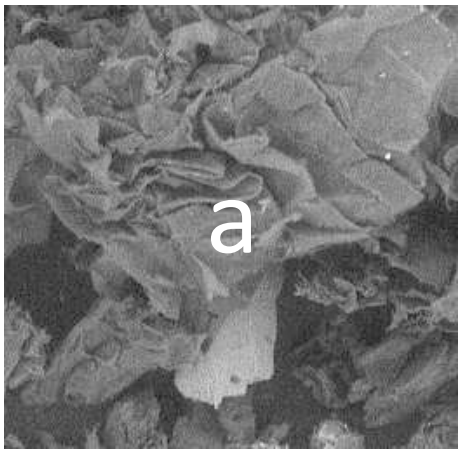
**2.6 Mechanical Evaluation**

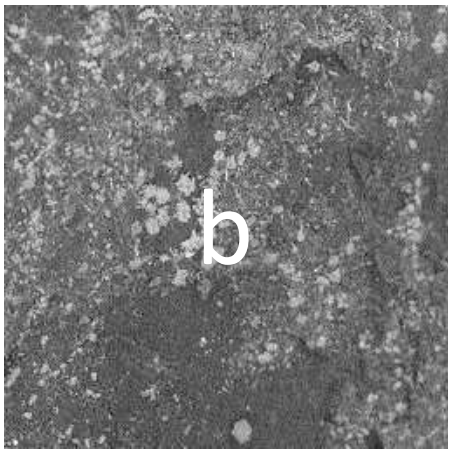
The following tests were further conducted as confirmatory tests on the compounded composites using standard test methods. Hardness (ASTM -D2240), abrasion resistance (ASTM-D5963-04), compressive strength (ASTM-D575-91), tensile strength, modulus, elongation at break and flexural tests (ASTM D3039/D). All measured properties were along the grain direction. Results are shown in table 2.

**3.0 Results and Discussions**

**3.1 Morphological Examination**

It could be seen that as carbonisation temperatures increase, the surface of the powder gets finer, clearer and an obvious optical clarity with the presence of tiny-like shinning particles could be observed. Darker stacked arrangements of aggregated particles are visible. The tiny glassy- like particle suggests regular arrangements, properly aligned morphologies and structure which could have resulted from modification. The more glassy and structured the particles are the better the interfacial interactions between filler and the rubber compound (Eiras and Pessan, 2009). The agglomerated areas create stres concentration zones which might act as hardness initiator and therefore leading to reinforcement in mechanical properties.



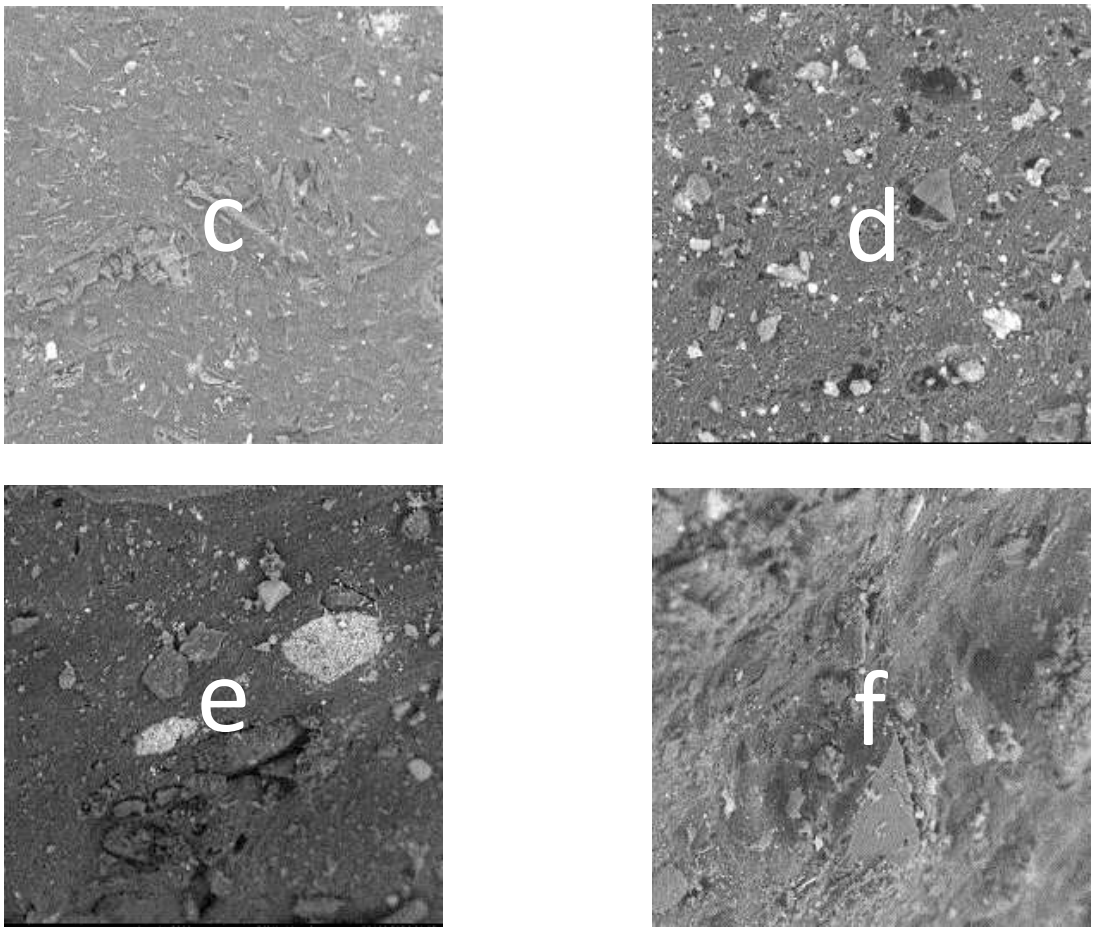



Figure 1: Microcellular views of coconut shell waste at (a) raw powder, (b) 300, (c) 400, (d) 500, (e) 600 and (f) $700^0$C

**3.2 X - Ray Diffraction**

As mathematically indicated in table 1 using a combination of the Bragg's Law (Eq. (1)) and Scherrer equation (Eq. (2)) the d-spacing, crystalline index and crystallite sizes (Popescu et, al, 2011) Percentage crystallisation were obtained from the diffractograms.

$$C.\ I = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \qquad (1)$$

Where, $I_{002}$ and $I_{am}$ are the intensities of the crystalline and amorphous regions. The apparent crystallite size (L) (Eq. (2)) was calculated using the Scherrer equation (Popescu et al, 2011):

$$L = \frac{k * \lambda}{\beta \ \ Cos\,\theta} \qquad (2)$$

Where K is a constant of value 0.9, λ is the X-ray wavelength (0.1542nm), β is the half-height width of the diffraction band and θ is the Bragg angle corresponding to the (200) plane.

**Table 1**: Crystalline Parameters

| **Parameter for XRD** | **CSP** | **300** | **400** | **500** | **600** | **700** |
|---|---|---|---|---|---|---|
| Crystallinity (%) | 60.2 | 71.2 | 82.9 | 87.7 | 86.2 | 85.6 |
| ACD ($A^0$) | ≈3.7 | ≈3.7 | ≈3.7 | ≈3.7 | ≈3.7 | ≈3.7 |
| Intensity Counts | 44 | 52 | 72 | 87 | 85 | 84 |
| FWHM | 0.20150 | 0.16520 | 0.16000 | 0.15970 | 0.15180 | 0.11820 |
| 2θMaximum Peak | 21.0759 | 22.0779 | 23.0465 | 24.9875 | 23.9441 | 23.0689 |

From table 1 above, as carbonisation temperatures increase, the amorphous regions tend to diminish compared to the stability of the crystalline region. As the amorphous region diminishes, the more stable crystalline region becomes more intensified, thereby leading to increase in percentage crystallinity (Popescu et al, 2011). The Average Crystalline Dimensions (ACD) are averagely the same possibly as the particle size of the powder were all at 100µm. Intensity counts increases with carbonisation temperature leading to more crystalline region up to the $500^0$C. Full width at half Maximum (FWHM) decreases with carbonisation temperature thereby reducing the d-spacing between particles and improvement on Crystallinity. The Bragg's angle (2θ) peaked within narrow range of values for the temperatures used; possibly around the characteristic peak value for the coconut shell (Momoh et al, 2016b). Increase in the percentage of crystallinity will increase the mechanical, chemical, optical and thermal properties of the coconut shell powder and consequently an indicative increase in the reinforcement properties of the vibration dampener (Eiras and Pessan, 2009; Joseph et al, 2003). Also see the diffractogrames of raw and at $500^0$C carbonisation treatment in figures 2 (a) and (b) below.

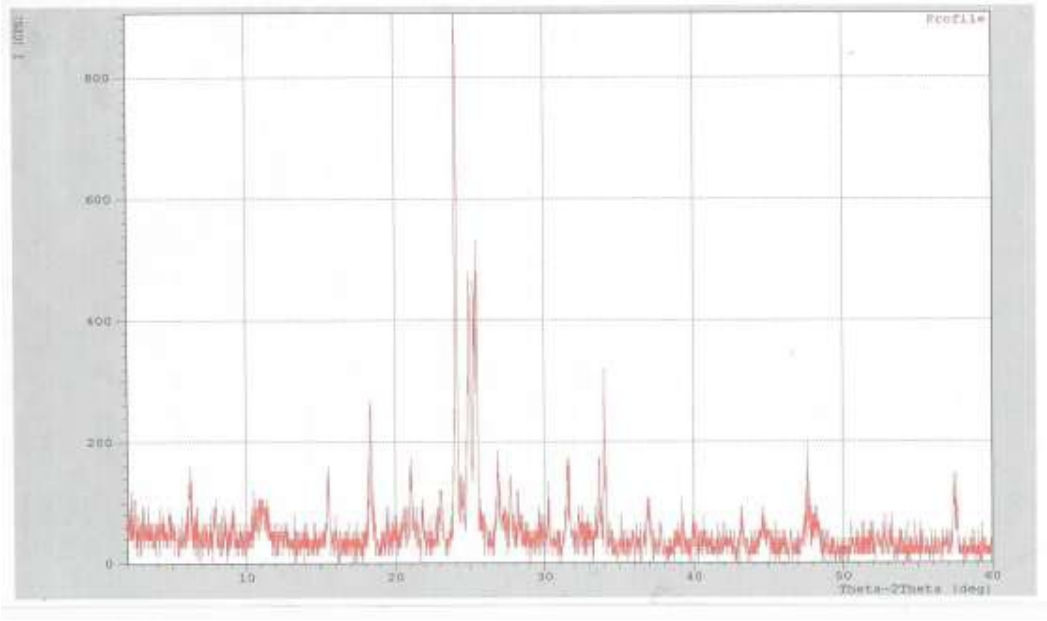
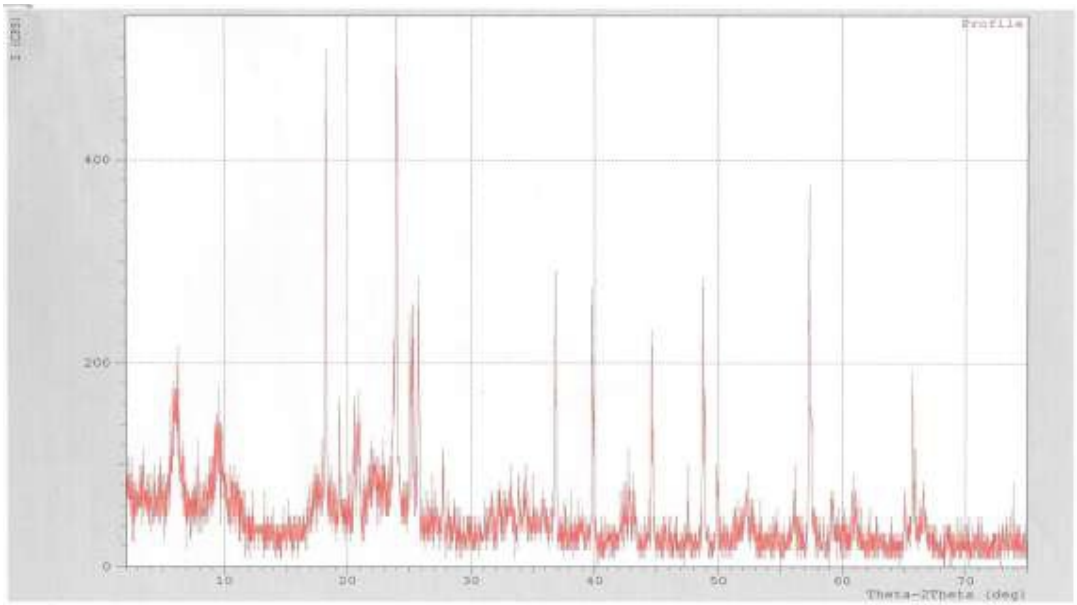



**Figure: 2a**




Figures 2 (a and b): X-ray diffractograms of raw and carbonised coconut shell powder waste at best temperature of 500ºC

**3.3 Mechanical Properties**

The results of the mechanical properties confirmed increase in hardness, abrasion resistance, tensile strength and modulus up to carbonisation temperature of $500^0$C. Possibly as a result of increase reactivity between the filler and the rubber matrix. These clear increases will definitely lead to increase in product reinforcement. The values of elongation at break and compression set decrease with increase in carbonisation temperature possibly due to the adherence of the filler to the polymer chain. The stiffening of the chain leads to high resistance to stretch when the strain is applied (Onyeagoro, 2012). Basic mechanical properties started decreasing at carbonisation temperatures above $500^0$C. See results on table 2 below.

**Table 2:** Mechanical Properties of the Composites

| Properties | CSP | 300 | 400 | 500 | 600 | 700 |
|---|---|---|---|---|---|---|
| Hardness (Shore A) | 73.00 | 75.00 | 78.00 | 86.00 | 80.00 | 76.00 |
| Abrasion Resistance | 22.51 | 28.42 | 32.10 | 36.90 | 44.84 | 37.42 |
| Compression set (%) | 28.90 | 26.40 | 24.80 | 22.30 | 23.00 | 23.20 |
| Tensile Strength (MPa) | 3.91 | 4.21 | 4.30 | 5.20 | 4.90 | 4.80 |
| Elongation @ Break (%) | 494.50 | 484.60 | 420.00 | 398.20 | 395.00 | 398.20 |
| Modulus (%) | 1.92 | 3.10 | 4.35 | 5.40 | 5.38 | 5.12 |
| Flexural Strength (MPa) | 0.99 | 0.39 | 0.28 | 0.46 | 0.38 | 0.21 |

**3.4 Fatigue and Resilience Test**

The crack initiation and resilience rebound analysis carried out on the designed component using the optimised formulation of coconut powder waste performed effectively well when compared to commercially available dampener in the market.

**Table 3** Crack initiation time and rebound resilience of the Automobile part (Motorcycle Dampener)

| Samples | Crack Initiation Time (Minutes) | Rebound Resilience |
|---|---|---|
| Raw Shell Waste | 44 | 60 |
| Carbonised Shell @ 500$^{o}$C | 130 | 55 |

**3.5 Pictorial View of Product and Mould**

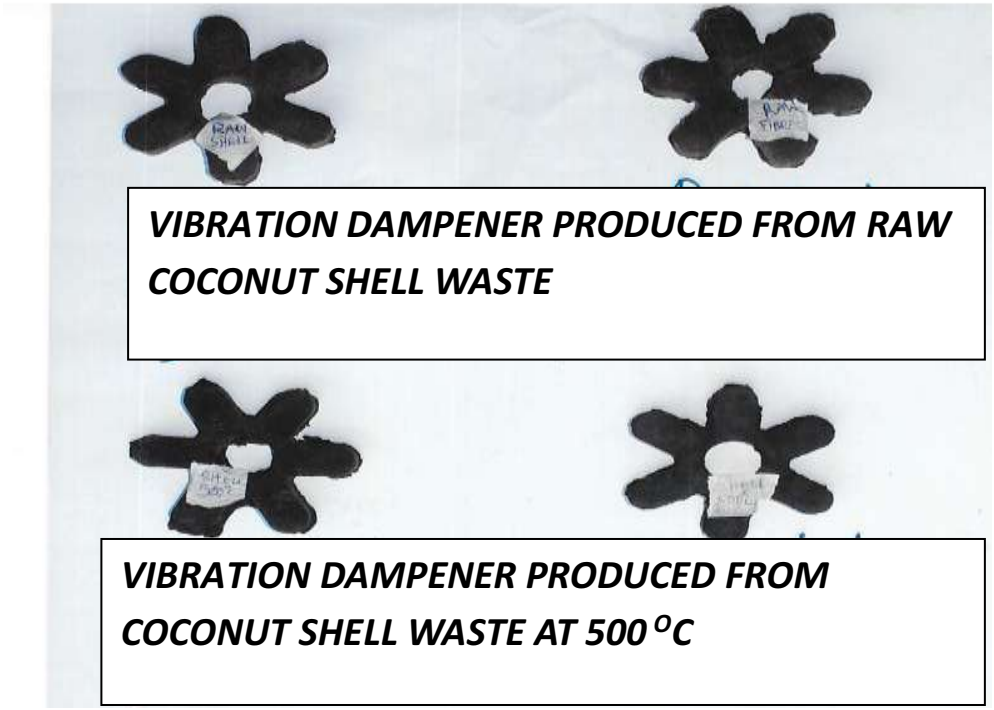



**Figure 3: Automobile part (Vibration Dampeners for Motor Cycle Hubs) from Optimised Formulation of Raw Coconut Shell Waste and Carbonised Shell at 500$^{o}$C**

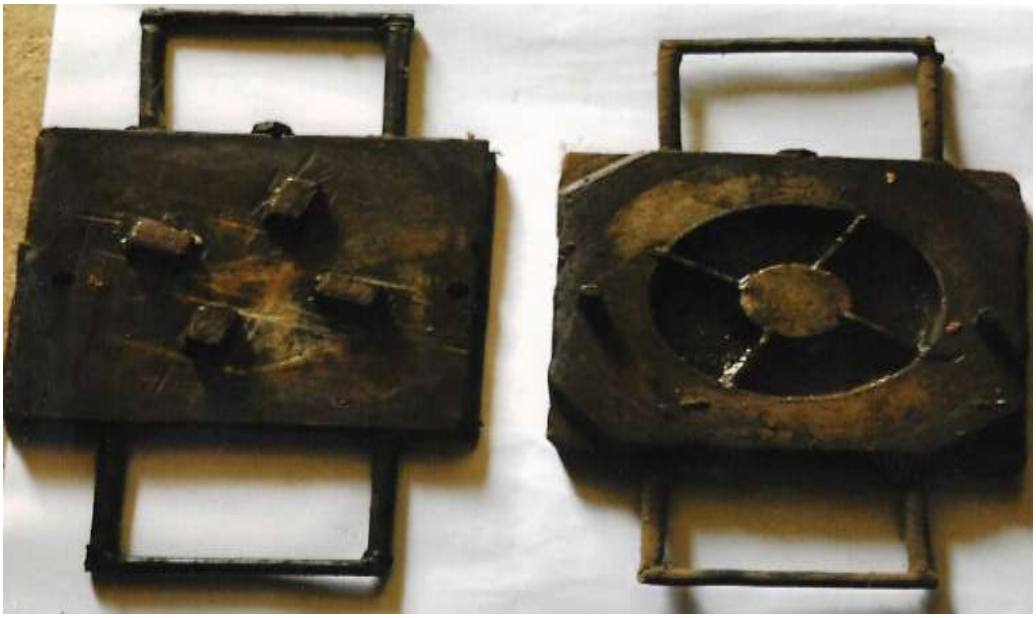


**Figure 4: Designed and Constructed Mould consisting of the Male and Female Parts used for the Compression Moulding Process of automobile part (Vibration Dampeners of Motor Cycle Hub)**

**4.0 Conclusion**

Modification through carbonisation diminished the amorphous region and intensified the crystalline region of the coconut shell waste powder. Morphological examinations also indicates finer and clearer particle aggregates and hence an improvement in filler-matrix interactions and reinforcements. Increase in finer particle aggregates, intensification of the crystalline region and decrease in crystallite size of the coconut shell powder became pronounced with increase in carbonisation temperature. There was a noticeable reduction of the full width at half maximum (FWHM) of the X-ray diffractograms, but the d-spacing were averagely the same.

However, the highest crystallinity and morphological built up occurred at the $500^0$C of carbonisation. Mechanical properties of hardness, tensile strength, abrasion resistance, and modulus were also more pronounced at $500^0$C of carbonisation. The inference here is that carbonisation of the coconut shell waste powder has a major effect on the crystalline intensity, morphological outlook and mechanical properties of the vibration dampener. These observed positive effects lead to appreciable reinforcement and therefore recommend ably significant to the development of engineering products.

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# Extraction and Characterization of *Lophira lanceolata* Seed Oil for Application on Leather

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**Abstract:** Seed oil is a vegetable oil that is obtained from seed (endosperm) of some plants, rather than the fruit (pericarp). Generally, oil is a liquid, non-polar chemical substance at room temperature with hydrophobic and lipophilic characteristics. The seed oil of Lophira lanceolata (Namijin kade) was extracted using solvent (n-hexane) method of extraction which gave rise to 49.10±0.10% oil yield, and the physico-chemical properties of the oil were analysed using standard official methods. The iodine value was found to be 74.19±0.64mg$I_2$/100g, this suggest that the oil contain mainly saturated fatty acids. Moisture content and ash content of the seeds were found to be 0.73±0.26% and 0.897±0.03% respectively. The refractive index of the oil was 1.347 at 40C. The peroxide value is 2.87±0.12mol.peroxide/Kg. The low peroxide value is an indication of high stability of the oil against further oxidation. The acid value of the oil (1.20±0.13mg/KOH/g of oil) was within the range given by the standard for fatliquor production which is an indication that the Lophira seed oil cannot undergo rancidity easily. The saponification value of the oil was found to be 178.30±0.96mg $KOHg^{-1}$. The FT-IR spectrum of the oil at various peaks were observed. The carbonyl functional group: –C=O was observed at 1744.4$cm^{-1}$ frequency and at frequency 3011$cm^{-1}$, -C=C- in unsaturated hydrocarbon was observed. Finally, the fatty acid fractions of the oil as analysed by the use of GC-MS were found to be Palmitic acid of 62.88%, Linoleic acid of 9.66% and Oleic acid of 4.90% and these fatty acids are peculiar to the synthesis of fatliquor. This oil can have useful properties in the manufacturing of soap, cosmetics and lubricants.

**Keywords:** Fatliquor, FT-IR, GC-MS, Lophira lanceolata seeds oil, Namijin kade.

## 1.0 Introduction

In leather manufacturing, conversion of hide and skin into leather requires several chemical and mechanical steps to remove non-collagenous matter in pre-tanning operation [1]. After the tanning operation, in order to produce good leather of desirable quality, the tanned leather is lubricated in a fatliquoring step [2]. Fatliquoring is one of the critical steps for most types of leather especially in garment and upholstery leather manufacturing. The incorporation of fatty matter into leather fiber via fatliquoring process enhance leather full and soft handle, flexibility, pliability and at the same time improves its mechanical properties [3]. In addition to separating the leather fibers from each other, oils and fats are introduced into the leather matrix in order to prevent the adhesion of fiber to promote good leather properties [4]. The most economical and environmentally friendly oil for sustainable production of fatliquor in the world oils market is the vegetable oil [5]. Nigeria is rich in vegetable oils such as Palm oil [6], Soya bean oil [7], ground nut oil [8], cottonseed oil and Sun flower oil to mention but few which can be used in fatliquor production. However, most of the fatliquors used in the Nigerian leather industry are imported from other countries and has invested huge foreign currency on imports

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[9]. *Lophira Lanceolata* seed oil can become imperatively one of the substitutes of vegetable oils used for fatliquor production because, it is mostly not consumed by humans. The oil has a potential properties for better fuel due to the presence of methyl ester derivative [10]. It also has good cosmetic and medicinal properties suitable for making soap. In traditional medicine the oil is used to treat dermatosis, toothache and muscular tiredness. Rubbing the skin with the oil prevents dryness [11]. *Lophira lanceolata* is known locally as "Namijin kande" in Hausa [12] Saktojé" in Fufulde and ''Kofia'' in Gbaya [13] is a common medicine plant in Sokoto State, Nigeria [14].

## 2.0 MATERIALS AND METHODS

### 2.1 Material

*Lophira lanceolata* seeds (Namijin-kade) was sourced from Asawe/Saminaka village, Katari in Kachia Local Government Area, Kaduna State, Nigeria, which was identified as *Lophira lanceolata*, belonging to Fabaceae family with in Department of Botany, Faculty of Life Science, Ahmadu Bello University, Zaria, Nigeria.

### 2.2 Oil Extraction

Lophira lanceolata seeds of 100.019g were crushed with mortar and pestle to powder form. Oil was extracted from the ground seeds with 350ml volume of n-Hexane for 1hour: 30minutes at 50ºC by the use of soxhlet apparatus. The solvent (n-Hexane) was evaporated over water bath for 1hour and then kept overnight until completely evaporated.

### 2.3 FT-IR Analysis

The functional groups of the oils were determined using FT-IR Agilent Technologies, Cary 630 machine at ABU Multi-user Laboratory Zaria, Kaduna State; Nigeria with spectrophotometer resolution of 8 and ranging from 4000-650.

### 2.4 Physico – Chemical Characterization of the Seed Oil

The oil extracted directly from *Lophira lanceolata* seeds were been analysed for their Saponification value, Iodine value, Peroxide value, Ash content, Acid value, Moisture content, Refractive index and pH value using Official Standard Methods described by [15][16][17].

### 2.5 Fatty acid composition determination

The crude *Lophira lanceolata* seed oil was analyzed to determine the fatty acids composition using Agilent GCMS 7890A coupled with MSD 5975C machine in American University of Nigeria, Yola; Adamawa State. There was not sample preparation method, it was run directly at running time of 21mins at temperature of 20ºC/min to 280ºC for 9mins.

## 3.0 RESULT AND DISCUSSION

### 3.1 Physical Properties

**Table 1: Physical Properties of Lophira Seeds/Oil**

| Parameter | Lophira seed/oil |
|---|---|
| Moisture content (%) | 0.73±0.26 |
| Ash content (%) | 0.897±0.03 |
| Oil yield (%) | 49.10±0.10 |
| pH | 6.47 |
| Refractive index (27$^{o}$C) | 1.347 |
| Solubility of oil in water | Insoluble |

The moisture content of *Lophira lanceolata* seeds is very low when compared to legumes with the required two years minimum storage moisture content of 11.0% [18][19]. This is indicative of high dry matter content, reduced antimicrobial activities, reduced redox reaction and high storage shelve life [20]. The percentage ash

content 0.897±0.03% indicates low inorganic fraction. The percentage oil yield 49.10±0.10% indicate that the seed contain much oil. The high percentage oil yield suggest commercial viability for Industries such as cosmetic [21], leather [22], soap, biodiesel [23] etc. The results obtained for the oil samples shown in Table 1 indicated that the pH value of *Lophira lanceolata* seed oil is 6.47. This shows that the Lophira seed oil is weakly acidic and almost neutral, implying that it contain low amount of fatty acids making it fit for consumption as compared to groundnut seed oil with ranged of values between 6.59 - 6.79 [24]. This implies that they contain low amount of fatty acids making them fit for edible purposes. The refractive index of the Lophira seed oil was 1.346 indicating the purity of the oil and the value obtained was in agreement with the standard limits set by CODEX.

**3.2 Chemical Properties**

**Table 2: Chemical Analysis Result of Lophira Seed Oil**

| Parameter | Values: Mean±SD | Units |
|---|---|---|
| Acid Value | 1.20±0.13 | mg/KOH/g |
| Iodine Value | 74.19±0.64 | $mgI_2/100g$ |
| Peroxide Value | 2.87±0.12 | mol.peroxide/Kg |
| Saponification Value | 178.30±0.96 | mg/KOH/g |

The results in table 2 shows the chemical analysis of the *Lophira lanceolata* seed oil for fatliquor production. Acid value is a measure of rancidity. It was stated that, if the acid values are high, the fat or oil will become more rancid and vice versa. For an oil to be potentially suitable as a raw-material for fatliquor synthesis, the acid value should be below 8.6 mg/KOH/g of oil [25]. It is observed from the results in table 1 above, that the average acid value for the oil extracted from *Lophira lanceolata seed* was 1.20±0.13mg/KOH/g, therefore the oil becomes imperatively suitable for fatliquor production. Saponification value of oil measure the average molecular weight of the triacylglycerols in a sample. Saponification value is inversely proportional to the mean molecular weight of the fatty acids that is, the smaller the saponification values, the larger the average molecular weight of the triacylglycerols. The saponification value: 178.30±0.96mg/KOH/g of *Lophira lanceolata* seed oil was found within the range given by [26]. The higher the Saponification value of the oil (178.30±0.96mg/KOH/g *Lophira lanceolata* seed oil), the smaller the molecular weight and therefore; the higher the penetrating powers of the oil into the leather, which gives it the softness properties after the fatliquoring processes. Iodine value is a measure of the degree of unsaturation. A low iodine value shows that the fat has a low quantity of unsaturated fatty acid [27]. The results in table 2 above indicated that Lophira seed oil has an Iodine value of 74.19±0.64$mgI_2/100g$ that agrees with the Iodine value range reported [28]. *Lophira lanceolata* seed oils with iodine value of 76.59$mgI_2/100g$. Fats and Oils are usually classified on the basis of their iodine values as drying oil (130 – 190), semi – dying oil (100 – 130) and non – drying (100 below) [29]. Hence, based on the iodine value of Lophira seed oil, it can be referred to as non – drying oil which can be used for the production of fatliquor. Peroxide value is a measure of the extent to which an oil sample has undergone primary oxidation. The peroxide value 2.87±0.12mol.peroxide/Kg from table 2 above indicated the very low level of hydroperoxides which could initiate or propagate further oxidation of the oil. The value was compared with the 3.0mol.peroxide/Kg peroxide value of rapeseed oil as reported by [30].

**Table 3: Fatty acid fractions of *Lophira lanceolata* seed oil**

| Fatty acid | Percentage (%) |
|---|---|
| Palmitic acid | 62.88 |
| Linoleic acid | 9.66 |
| Oleic acid | 4.90 |
| 13-Octadecenoic acid | 1.85 |
| Cis-Nonadecenoic acid | 0.87 |
| Cis-Vaccenoic acid | 0.10 |

Fatty acid compositions of the *Lophira lanceolata* seed oil extracted is shown in Table 3. As can be observed, Palmitic acid, Linoleic acid and Oleic acid were the main fractions and were found present with the following percentages respectively: 62.88%, 9.66% and 4.90%. These constituents account for above 75% of the fatty acids. The saturated fraction account for over 60% and the unsaturated fractions account for less than 16% of the total fractions.

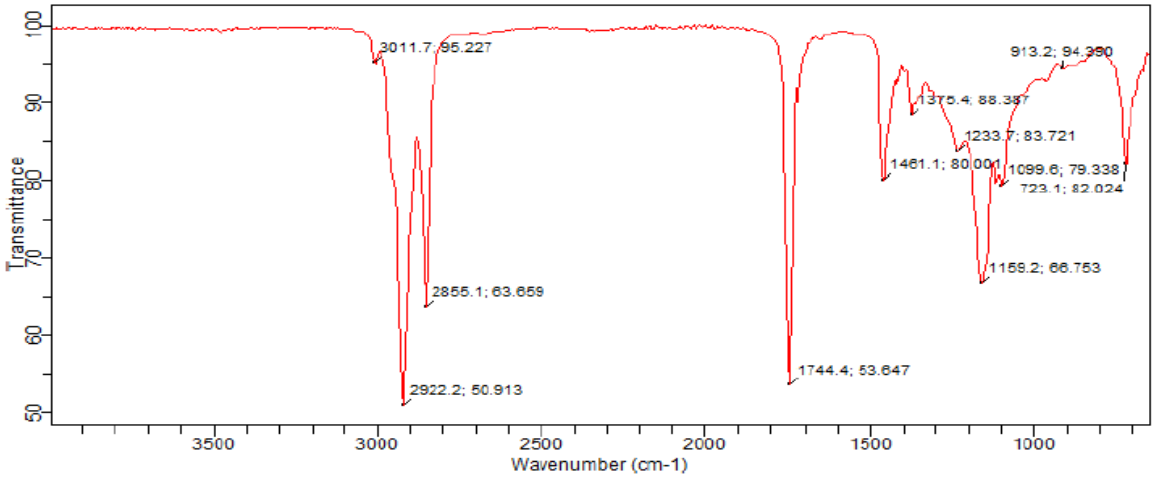


**Figure 1: FT-IR Analysis of the Raw *Lophira lanceolata* Seed Oil**

The peaks at figure 1: 1744cm$^{-1}$ and 3011.7cm$^{-1}$ are due to the presence of –C=O and –C=C- respectively. With the presence of the unsaturated functional group, indicated that the oil is capable of producing fatliquor [31].

## 4.0 Conclusion

The crude extract of *Lophira lanceolata* seed oil examined in this work by its saponification value, pH value, peroxide value, acid value, iodine value and refractive index. The infrared spectroscopic and GC-MS analysis shows the level of Unsaturated and Saturated fatty acids which are ingredients for the manufacturing of fatliquor. Thus, the results indicated that, the Lophira seeds oil is imperatively viable for the synthesis of fatliquor. They may however be useful for other purposes such as soap, judging by their high saponification values in the range of 178-261 mg KOHg-1.

**Acknowledgements**

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# TREATMENT OF LEAD CONTAMINATED WASTEWATER USING ALUMINIUM SULPHATE AND MORINGA OLEIFERA AS COAGULANTS

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**ABSTRACT:**
*Treatment of lead-contaminated water using aluminium sulphate and* Moringa *oleifera as coagulants has been studied. Proximate analysis on the moringa oleifera seed showed that it has a high protein content of 32.57%, whereas moisture and ash contents were 6.82% and 6.02%.This showed that moringa olefera performed better than aluminium sulphate because of it's higher percentage of protein content. In addition, the crude fibre and fat contents were 17.5% and 29.44% respectively. The Fourier Transform Infra-Red of (Agilent Technologies CARY 630 model) was used. The spectra revealed several functional groups of ≡C-H stretch of alkynes,C=O stretch of ester, N-H bend of amides and =C-H bend of alkenes which were naturally present in the moringa oleifera. In thecoagulation-flocculation process, effects of five selected parameters were studied as they ultimately have an effect on the process. They were effects of pH which showed that both moringa oleifera and aluminium sulphate has better pH for water treatment. Settling time for the two coagulants were good this aids for their good performance. Aluminum sulphate has a higher removal of 71.36% at $60^0$C while moringa olerifera has 64.54% removal at $60^0$C. Moringa olefera has optimum dosage of 2.00g with 71.59% as the percentage removal while aluminium sulphate has 2.50g with 74.99% as the percentage removal. The optimum lead removal at the concentration of 14mg/L for the both coagulants were 71.42% for moringa oleifera and 75.01% for aluminium sulphate. From the investigation, moringa oleifera is better than aluminium sulphate because it is a natural polymer with no harm to human health , environmental friendly and available in the market and can be planted as an economic tree in Nigeria. Aluminium sulphate cannot be compared to a natural substance like moringa oleifera in performance*.
**Keywords:** *Wastewater Proximate analysis, Coagulation, Flocculation*.

## 1.0 INTRODUCTION

Potable water supply is a basic need required for living creatures and human being specifically. Developing countries and third world countries are facing potable water supply problems because of inadequate financial resources (Arnoldsson, Bergman, Matsinhe& Persson, 2008). The pervasive utilization of water by today's industries (with reference to paint industries) has brought about a severe problem of drainage and disposal of industrial wastewater. Effluents from these industries are degrading the underground and surface water quality through seepage and discharge into rivers, due to toxic and undesirable chemical constituents present in them and thus, being a major cause of water pollution (Matilainen, Lindqvist &Tuhkanen, 2005). The constituents of ―this wastewater are of inorganic, organic and toxic nature and require extensive treatment before discharge to prevent physical, chemical and biological pollution of the scarce water resources. Mahmood and Malik, (2014) reported that the untreated paint effluents are harmful to human beings when

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disposed without adequate treatment as they contain a lot of pollutants such as heavy metals.

Heavy metals have been found to be highly toxic especially when their natural concentrations are exceeded. At normal concentration, they promote the functions of the enzymes but could lead to a lot of adverse metabolic reaction when their concentration rises beyond tolerance limit. This massive quantity of untreated effluent is disposed to the river side and cause considerable load on the water courses leading to widespread damage to aquatic life and to the environment. Lead, cadmium, chromium, nickel and copper are particularly common heavy metals found in paint industrial wastewater. Lead contamination of the environment is primarily due to anthropogenic activities making it the most ubiquitous toxicmetal inthe environment. Lead readily accumulates in the humus-rich surface layer of the soildue to its complexity with organic matter and it was reported to be the least mobile heavy metal insoils under reducing or non-reducing conditions. It hasbeen the most common heavy metal contaminant inurban soil due to atmospheric depositions fromautomobile emissions and industries.

Huge amount of research has already been carried out on effluent treatment using different technologies, such as flotation, electrochemical treatment, sedimentation, coagulation, filtration, ultrafiltration and reverse osmosis process (Krishanamoorthi, Sivakumar, Saravanan, &Prabhu, 2008; Song, Williams, &Edyvean, 2004). In addition, the cost of water treatment is increasing,and the quality of available water is not stable due to suspended and colloidal particle load caused by land development and high storm runoff during the rainy seasons (Mahmood & Malik, 2014). During the rainy seasons, the contaminant level increases and the need for water treatment chemicals increase as well, which leads to high cost of treatment which the water treatment companies cannot sustain (Arnoldsson et al., 2008). As a result, the drinking water that reaches the consumer is not properly treated. Amongst other treatment techniques, coagulation has been known to man for effluent treatment since the 19th century (Genovese & González, 1998).

Coagulation involves the addition of chemical coagulants to destabilize particles, and flocculation involves the contacting of destabilized particles with the intent of forming agglomerations of particles (flocs) that are more amenable to solid-liquid separation (Reynolds & Richards, 1996). In practice, the distinctions between coagulation and flocculation are often blurred (Benjamin & Lawler, 2013). Coagulation and flocculation are usually followed by sedimentation, filtration, and disinfection, in the primary stage, succeeded by chlorination. This method is used worldwide for water treatment before it is finally distributed to the consumers. Various types of coagulants are used in typical water treatment processes for making the water fit for use by the consumers. These can be classified into inorganic coagulants, synthetic polymers, and biological coagulants.

Coagulants originating from vegetables and seeds were in use for the purpose of water treatment before the wide scale use of chemical salts, but they have not been able to displace the use of chemical salts as the scientific grasp of their effectiveness and mechanism of action was lacking (Wani, Hasan & Malik, 2010). The utilization of biological coagulants has not picked up so far because of the lack of clarity in the method to use them commercially. They have given way to salts, typically aluminum sulphate, progressively under modernization and survived only in some parts of some developing countries (Binti-saharudin&Nithyanandam, 2014).

The commonly used metal coagulants fall into two general categories: those based on aluminum and those based on iron. The aluminum coagulants include aluminumsulfate, aluminum chloride and sodium aluminate. The iron coagulants include ferric sulfate, ferrous sulfate, ferric chloride and ferric chloride sulfate. Other chemicals used as coagulants include hydrated lime and magnesium carbonate. Moringa Oleifera, a natural coagulant, can be used in place of artificial coagulant in the treatment of wastewater. Moringa oleifera (MO) seed is a natural plant with active bio-coagulate compounds that can be used for water clarification since it reduces the use of chemical-based coagulants. The water soluble extract of the seeds contains a cationic protein which has been proven to have a dramatic coagulation effect on suspended and dissolved particles in highly turbid water.

## 2.0 MATERIALS AND METHODS

### 2.1 Materials

Moringa Oleifera obtained from Abakpa market in Enugu State was the raw material used in this study as natural coagulant. The analytical grade chemicals (Kemwl) used include aluminium sulphate, lead nitrate, ,hydrochloric acid and sodium hydroxide.

### 2.2 Proximate Analysis of the Samples

The procedures described by the AOAC were used in carrying out the proximate analysis. Thiswas doneso as to determine the percentage composition of the sample. The parameters checked for include: moisture content (%), ash content (%), crude fibre content (%), fat content (%) and protein content (%).

### 2.3Collection and Simulation of the Wastewater Samples

Effluents from a Efet paint industries, Agbara Ogun State, was collected in four litres plastic container. The concentration of this effluent was checked using the AAS. The synthetic wastewater contaminated with lead was hence prepared,and concentration also analysed. To simulate the contaminated wastewater, a known mass of lead metal salt was incorporated into 1000 ml of the distilled water. Thiswas observedto simulate standard (wastewater) solution for treatment using the coagulant types respectively. The initial and final concentration of the solution as detected by the spectrophotometer was recorded.

### 2.4 Fourier Transform Infrared (FTIR) Spectroscopy

The Fourier transform infrared (FTIR) spectrum of the coagulant was used to determine the vibration frequency changes of the surface functional groups on the sample. This helped to identify the functional groups that were active in the coagulation-flocculation processes.

### 2.5Coagulation-Flocculation of Simulated Wastewater

A number of factors can affect the coagulation–flocculation process: pH, settling time, temperature, coagulant dosage and concentration of the target pollutant. These factors individually and collectively have a great influence on the coagulant's optimum performance. The factors were investigated keeping the synthetic effluent volume constant at 50ml in 250-mL Erlenmeyer flasks. The five selected factors were thus investigated.

## 3.0RESULTS AND DISCUSSION

### 3.1 Proximate Analysis

The results of the proximate analysis of the sample are shown in Table 3.1. As shown in the table, the Moringa Oleifera seed has a high protein content of 32.57%, whereas moisture and ash contents were 6.82% and 6.02%. In addition, the crude fibre and fat contents were 17.5% and 29.44% respectively. Alessandro et al., (2016) reported a protein, moisture and ash contents of 31.4%, 7.0% and 6.2% respectively. The analysis helps in understanding the specific seed storage

protein responsible for coagulating ability (Oliveira, Silveira, Vasconcelos, Cavada, & Moreira, 1999; Santos, Argolo, Coelho & Paiva, 2005).

**Table 3.1: Proximate Analysis of Moringa Oleifera**

| Parameters (%) | Moringa Oleifera |
|---|---|
| Moisture Content | 6.82 |
| Ash Content | 6.02 |
| Crude Fibre Content | 17.5 |
| Fat Content | 29.44 |
| Protein Content | 32.57 |

### 3.2 FTIR Spectrum

Figure 3.1 shows the spectrum of Moringa Oleifera. The FTIR is a powerful technique for the interpretation of structural changes in samples. The absorption peak at 3283.8 $cm^{-1}$ represents the ≡C-H stretch of alkynes whereas alkanes and alkyls were represented by the peak at 2922 and 2855 $cm^{-1}$. The 1744.4cm-1 represents the C=O stretch of esters whereas O=C-O-C stretch at 1230$cm^{-1}$ represents another class of ester. The absorption at 1651.2 and 1543.1$cm^{-1}$ represent the C=O stretch and N-H bend of amides. Presence of =C-H bend which is typical of alkenes was made evident by the bands at 875.9 and 783.9$cm^{-1}$ respectively. Similar results have been confirmed by other researchers (Marcus &Nwineewii, 2015; Packialakshmi& Archana, 2014).

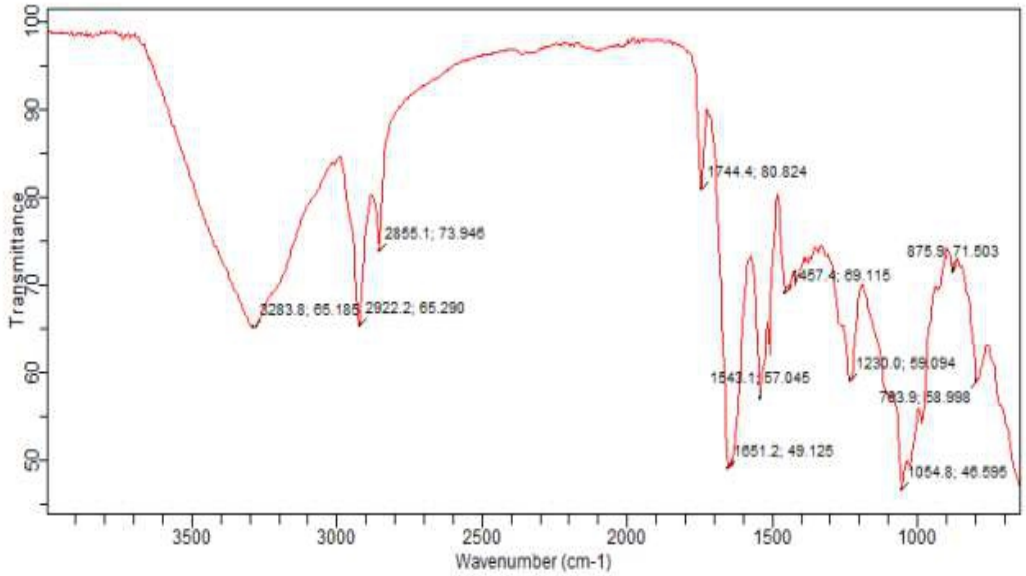


**Figure 3.1: FTIR Spectrum of Moringa Oleifera**

### 3.3 Coagulation-Flocculation Studies

In the coagulation-flocculation process, it is important to control certain parameters as they ultimately have effect on the process. The parameters studied were effects of pH, settling time, temperature, coagulant dosage and initial metal ion concentration respectively.

#### 3.3.1 Effect of pH on the coagulation-flocculation of the lead metal ion.

In this study, pH was varied within the range of 2-10. The data illustrated in Table 3.2 and Figure 3.2 show the dependence of the process on pH for the two types of coagulant used. For both coagulants, at low pH (2), the percentage removals were 13.34% (aluminium sulphate) and 26.41% (Moringa oleifera). These later increased to maximum values at pH 6 (aluminium

sulphate- 45.70%) and pH 8 (Moringa oleifera- 49.28%). On attaining these optimum pH values, there was another fall off in elimination efficiency at high pH. Abdul and Azhar, (2013) reported an optimal pH for PAC was around 6-7 with COD removal of 58%. According to their report, this happened because the poly nuclear species were already present in this coagulant and the polymeric chain had been partially hydrolysed. Another report obtained an optimal pH 6 for Moringa oleifera (Ndabigengesere, Narasiah, & Talbot, 1995). They asserted that the amino acids present in M. Oleifera (cationic coagulant) get ionized and produce carboxylate and H+ charge which attract colloidal particles in the medium which get neutralized and settle down as flocs.

**Table 3.2: Effect of pH on the coagulation-flocculation of the lead metal ion.**

| pH | Percentage Removal, I (%) | |
|---|---|---|
| | Aluminium Sulphate | Moringa Oleifera |
| 2 | 13.34 | 26.41 |
| 4 | 27.29 | 35.77 |
| 6 | 45.70 | 42.29 |
| 8 | 35.72 | 49.28 |
| 10 | 20.57 | 28.61 |

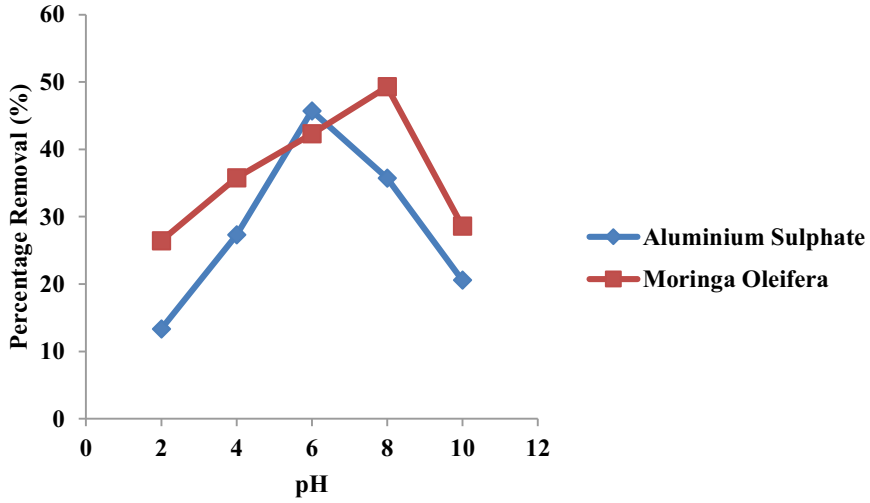


**Figure 3.2: Plot of percentage removal of lead metal ions at varying ph readings.**

**3.3.2 Effect of settling time on the coagulation-flocculation of the lead metal ion.**

Table 4.3 and Figure 4.3 show the settling time effect on the coagulation process at optimum pH values for both coagulants. The upturn in percentage removal with an increase in initial settling time (i.e. 15mins) was observed. For the aluminium sulphate, the optimum settling time was at 45mins with percentage removal of 53.56%. For the moringa oleifera, however, optimum settling time was at 60mins, with 57.05% as percentage removal value. On exceeding these optimal times, the percentage remove decreased because all colloids have already been neutralized and have got precipitated above the optimum time. On comparison both coagulants, moringa oleifera gave a higher optimum result than the aluminium sulphate. This shows the better efficiency of the natural coagulant over the artificial. The observation has been confirmed by other researchers using other target pollutants (Miller, Fugate, Craver, Smith, & Zimmerman, 2008; Nkurunziza, Nduwayezu, Banadda, &Nhapi, 2009; Pritchard, Craven, Mkanda wire, Edondson, &O'neill, 2010).

**Table 3.3: Effect of settling time on the coagulation-flocculation of the lead metal ion.**

| Settling Time (mins) | Percentage Removal, I (%) | |
|---|---|---|
| | Aluminium Sulphate | Moringa Oleifera |
| 15 | 45.73 | 49.39 |
| 30 | 48.40 | 50.76 |
| 45 | 53.56 | 53.74 |
| 60 | 51.44 | 57.05 |
| 75 | 49.87 | 55.56 |

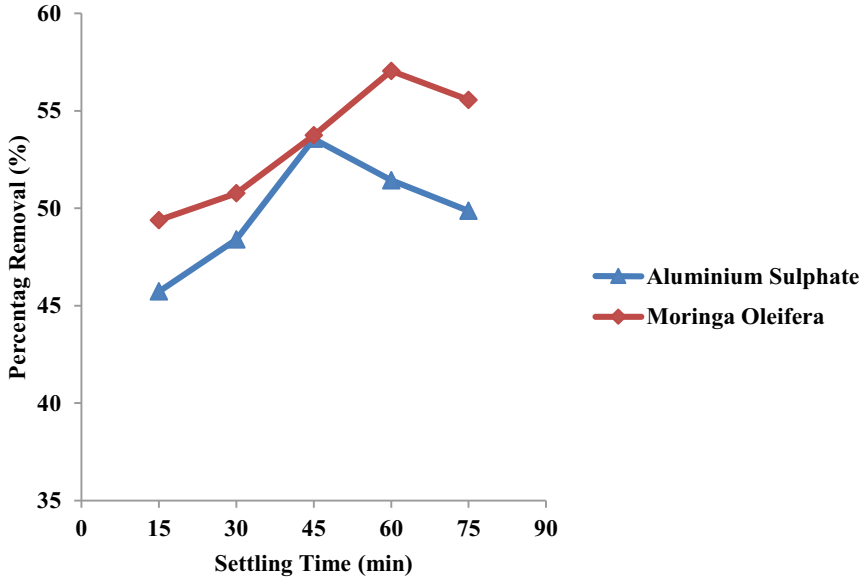


**Figure 3.3: Plot of percentage removal of lead metal ions at varying settling times**

**4.3.3 Effect of temperature on the coagulation-flocculation of the lead metal ion**

From Table 4.4 and Figure 4.4, it can be observed as with an increase in temperature from 30-40°C (i.e. aluminium sulphate), the percentage removal increased from 53.60-69.29%. On increasing the temperature to 50°C, a decrease in the removal efficiency was observed (i.e. 64.54%). This decrease continued till the last temperature range studied. For the moringa oleifera coagulant, the percentage removal was 57.11% at 30°C. This increased steadily to 71.36% at 60°C before a decrease of 62.94% was observed at 70°C. Analysing the data obtained in this work, moringa oleifera presented the best removal efficiency when compared with aluminium sulphate. Moringa oleifera has a high concentration of organic matter, nutrients and vitamins (Ghebremichael, Gunaratna, Henriksson, Brumer, &Dalhammar, 2005). These natural constituents could have increased its stability to a temperature increase during the experiment. In addition, the seed consists of a large amount of protein, and the active portion of this coagulant is linked precisely to the presence of a positively charged cationic protein with high molecular weight, which destabilizes particles that are present in water and promote the coagulation of colloids (Carvalho, Alves, Silva, Bergamasco, Coral, &Bassetti, 2016).

**Table 3.4: Effect of temperature on the coagulation-flocculation of lead metal ion.**

| Temperature (°C) | Percentage Removal, I (%) | |
|---|---|---|
| | Aluminium Sulphate | Moringa Oleifera |
| 30 | 53.60 | 57.11 |
| 40 | 69.29 | 60.79 |
| 50 | 64.54 | 63.56 |
| 60 | 61.14 | 71.36 |
| 70 | 59.27 | 62.94 |

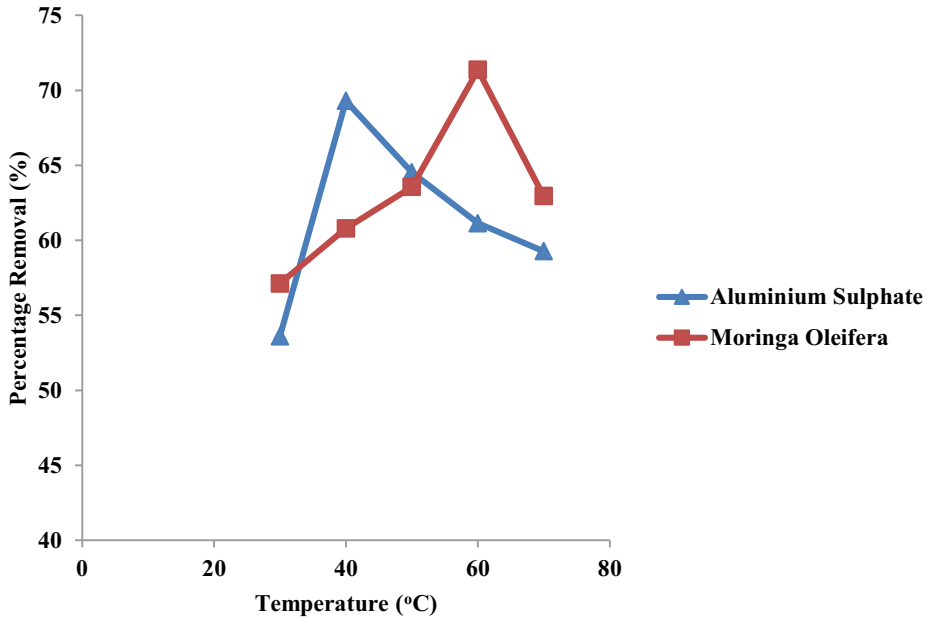


**Figure 4.4: Plot of percentage removal of lead metal ions at varying temperatures.**

**3.3.4 Effect of coagulant dosage on the coagulation-flocculation of lead metal ion.**

In evaluating the effect of the coagulant dosage on the process, optimum pH, optimum settling times and optimum temperature were used. The optimal dose of the coagulant is defined as the value above which there is no significant increase in removal efficiency with further addition of coagulant. Table 4.5 and Figure 4.5 clearly illustrate this effect studied. For the aluminium sulphate, optimum dosage was 2.00g with 71.59% as the percentage removal. On increasing the dosage to 2.50g, it decreased to 71.03%. According to Huang and Pan, (2002), at lower coagulant dosage, the only mechanism for destabilization of particles is charge neutralization. The reason behind this trend is that as soon as the coagulant concentration exceeds the optimum dosage, the excess coagulants will simply get added leading to increase in viscosity in water (Lee, Hanafiah, Halim, & Mahmud, 2015). This claim is valid since they did not interact with oppositely charged colloidal particles as those particles have already been flocculated earlier. However, a high concentration of polyelectrolyte forms an envelope on the suspending particles and causes them to remain in suspension, thus removal efficiency decreases (Demirci, Erdogan, &Ozcimder, 1998). For the moringa oleifera, the optimum dosage was 2.50g with a percentage removal of 74.99%. Hence, moringa oleifera coagulant

performed better than the aluminium sulphate used. According to some researchers, this can be attributed to the availability of more flocculating sites on increasing its dosage (Azhar, Ghanley, Suhardy, Farizui, & Irfan, 2005; Esmaeili, Ghasemi, &Rustaiyan, 2008).

**Table 3.5: Effect of coagulant dosage on the coagulation-flocculation of lead metal ion.**

| Coagulant Dosage (g) | Percentage Removal, I (%) | |
|---|---|---|
| | Aluminium Sulphate | Moringa Oleifera |
| 0.50 | 69.27 | 71.33 |
| 1.00 | 69.36 | 71.44 |
| 1.50 | 70.50 | 72.84 |
| 2.00 | 71.59 | 74.91 |
| 2.50 | 71.03 | 74.99 |

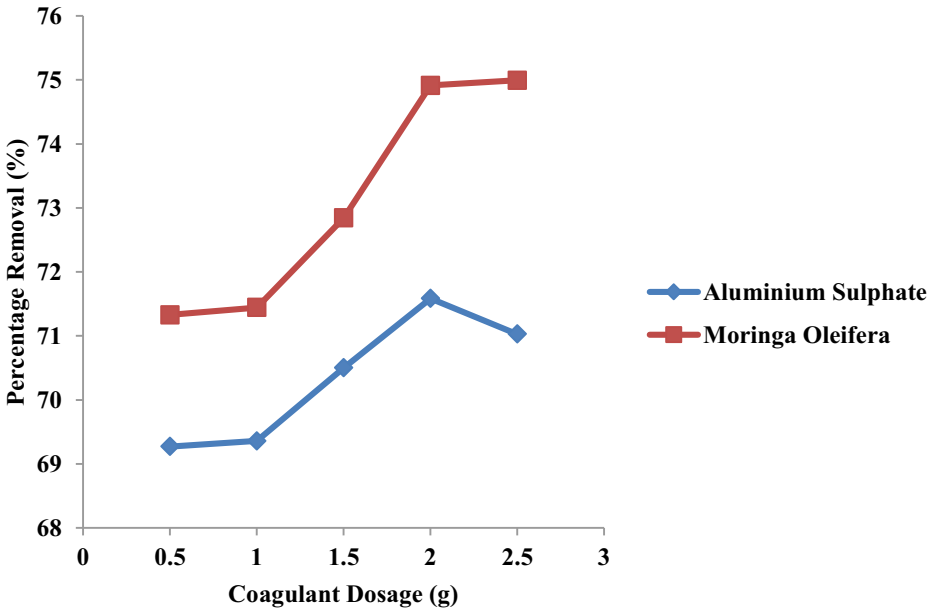


**Figure 3.5: Plot of percentage removal of lead metal ions at varying coagulant dosages.**

**3.3.5 Effect of initial metal ion concentration on the coagulation-flocculation of lead metal ion.**

In this test, the optimum parameters obtained previously were selected. The concentration of the lead ion was varied from 140-220mg/L for the two coagulants studied. The percentage removals versus initial metal ion concentration for each both coagulants are represented in Table 4.6 and Figure 4.6 below. The optimum lead removal was obtained at the concentration of 140mg/L for both coagulants. For aluminium sulphate, 71.42% removal was obtained while for moringa oleifera, 75.01% removal was obtained. Ping, Kamaruddin, and Mukthar, (2001) reported a 99% removal using alum. They asserted that the higher efficiency of coagulation process was observed due to higher sulphate contents in the solution as the transformation from $Al^{3+}$ to $Al(OH)_{3(S)}$ and precipitation were fast. Moringa oleifera was selected to be the most efficient coagulant since it removed a greater percentage of lead in the synthetic wastewater.

**Table 3.6: Effect of initial metal ion concentration on the coagulation-flocculation of lead metal ion.**

| Initial Metal Ion Concentration (mg/L) | Percentage Removal, I (%) | |
|---|---|---|
| | Aluminium Sulphate | Moringa Oleifera |
| 140 | 71.42 | 75.01 |
| 160 | 65.01 | 67.68 |
| 180 | 62.71 | 62.04 |
| 200 | 57.00 | 59.46 |
| 220 | 55.81 | 57.77 |

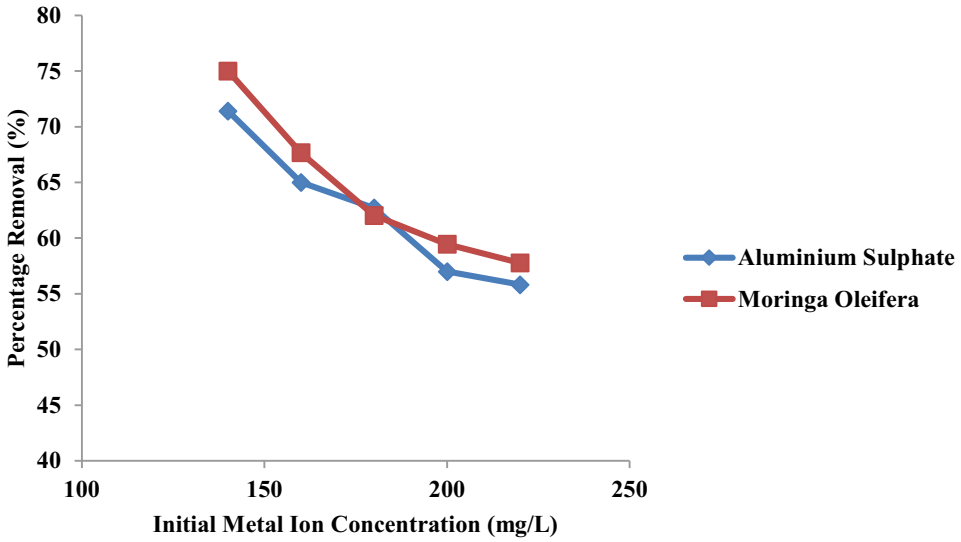


**Figure 3.6: Plot of percentage removal of lead metal ions at varying concentrations of lead ion.**

## 4.0 CONCLUSION

From this study, it was evidenced that both aluminium sulphate and moringa oleifera were good coagulants for the treatment of lead contaminated wastewater. However, the optimum removal efficiencies of moringa oleifera were continuously higher than the aluminium sulphate used. Hence, it stands as real alternative to aluminium sulphate coagulant in the wastewater treatment. It also presents the functional working advantages that may encourage further studies with regard to purifying and refining the active coagulant principal.

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