Extraction and Physicochemical Characterization of Cashew Nut-Shell Liquid in Metal Forming

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ABSTRACT: Effective lubrication during metalworking is critical to mitigating undesirable tool wear and poor surface finish of formed components. This paper investigated the physicochemical characteristics of cashew nut-shell-liquid (CNSL) with a view of determining its suitability for the development of an eco-friendly lubricant used in metal forming. The cashew nut shell oil (CNSO) was extracted by soxhlet method and characterised to determine its physicochemical properties including density, free fatty acid (FFA), and viscosity. In addition, the saponification value, iodine value, pH, flash point and friction coefficient were determined using relevant analytical tools. The results showed that CNSL yield from soxhlet solvent extraction is 45.6%, which is adjudged to be relatively substantial with regard to other extraction processes. Furthermore, the CNSL exhibited critical lubrication characteristics in terms of density (0.94 g/cm^3), moisture content (3.96%), acid value (11.32 mg.KOH/g), FFA (3.7%), ester value (127.92 mg.KOH/g), saponification value (139.24 mg.KOH/g), pH value (4.61), iodine value (40.21 gI_2/100 g), viscosity at 100 ^oC (14.12 Pa.s), friction coefficient (0.001) and flash point of 286.63 ^oC. Given these outcomes in comparison with commercial Chevron soluble oil, it is concluded that CNSL is suitable for use as a viable metal forming lubrication fluid.

KEYWORDS: Cashew nut-shell, Liquid, Lubrication, Metal forming, Physicochemical, Soxhlet extraction.

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I. INTRODUCTION

Development of varied lubricant blends from biowaste and other sources continue to attract attention owing to the need for efficient lubrication of metal forming tools. This stemmed from the knowledge that certain agricultural wastes are known to possess substances that are desirable in the production of quality oil suitable for friction reduction application (Mubofu, 2016; Fapohunda *et al*, 2021). Cashew nut shell (CNS) is one of such waste that if appropriately processed has potential application in fixing tribological phenomena challenges (Jabbarzadeh, 2018). According to Aina *et al* (2018), CNS thickness is about 3.2 mm and contains a dark reddish brown viscous liquid which has a bulk density of 481.8 kg/m^3.

Given the above description of CNS, it is safe to assert that, there is a high potential industrial application of CNSL if properly processed. Furthermore, the huge abundance of cashew nut in many southern states in Nigeria (Kogi, Oyo, Osun, Anambra, Imo and Enugu) and Africa generally necessitates the need to develop processes to recycle the huge tonnages of waste likely to be generated from cashew processing (Mwangi *et al,* 2013; Kyei *et al,* 2019; Sekunowo *et al,* 2021). For example, as at 2019, Nigeria produced about 100,000 metric tons of cashew nuts representing 12% of the world 835,000 metric tons (Agada and Sule, 2020; Adesanya *et al*, 2021). About 90% of the local production is exported in form of raw nuts (Oluyole *et al*, 2017).

The processing of CNS entails extraction of the liquid (oil) in the shell and characterising it for its physicochemical parameters. Thus, several CNSL extraction methods have been developed which are widely reported in literature. These methods among others include pyrolysis (Patel *et al*, 2006), thermo-mechanical or hot oil-bath (Tyman *et al*, 1989; Ghandi *et al*, 2013), screw press (Himabindu *et al*, 2015) and soxhlet extraction (Subbarao *et al*, 2011). Of the array of these extraction methods, the screw press route is adjudged most appropriate on industrial scale while the solvent method is known to produce quality extract (Himabindu *et al,* 2015). Furthermore, the expeller route of CNSL extraction is both economically and technologically suitable for instant application (Mubofu, 2016).

Through physicochemical analysis, CNSL is established to contain about 90% anacardic acid by weight (Kubol *et al*, 2006; Morais *et al*, 2017). However, salicylic acid being one of the derivatives of anacardic acid has been shown to easily decompose to cardanol on heating (Akinhanmi *et al*, 2008). The balance of 10% CNSL contains cardol, a resorcinol derivative with long unsaturated hydrocarbon chain (Risfaheri *et al*, 2009). As reported by Azizian and Khosravi (2019), cardanol also exhibits a type of chemical structure that is analogous to that of phenol thus, it has the potential to replace phenol. These bio-substances are capable of functioning as oxidation agent in lubricating blends which enhance lubrication property stability at elevated temperatures.

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Efficient lubrication during metal forming provides the means by which frictional force is substantially reduced thereby enhancing the tool-life. In addition, lubrication is meant to cool and maintain the tool and work-piece at ambient conditions. This will ensure accuracy of formed component's dimensions which often proof difficult to achieve due to thermal expansion giving rise to poor surface finish (Diniz and Micaroni, 2002). Although, conventional metal forming fluids are known to be petroleum based. However their application has attracted stiff opposition from machine operators owing to its debilitating health hazards and collateral environmental challenges (De-Chiffre, 1988). Consequently, replacement of petroleum-based lubricants with those derived from vegetable oils is a current research focus. It is envisaged that this will also significantly reduce the greenhouse effects and enhance recyclability of most agricultural wastes. The above formed the motivation for the current study which is aimed at determining the suitability of CNSL as a viable lubrication fluid in metalworking.

II. MATERIALS AND METHODS

A. Materials

The major materials employed for this research include cashew nut shells (Figure 1) sourced from a cashew processing firm located in Sango-Ota, Ogun State, Nigeria and n-hexane solvent sourced commercially at Ojota, Lagos, Nigeria. CNSs were crushed mechanically using a milling machine (Figure 2). This was done to increase the surface area thereby enhancing reactivity with the n-hexane solvent used for the extraction. The crushed CNS was then kept at room temperature in a jar to prevent fermentation.

Figure 1: Cashew nut-shells.

Figure 2: Customized milling machine.

B. Methods

1) Extraction of CNSL by soxhlet process

The extraction of CNSL was accomplished using a soxhlet extractor model, EX5/75, QUICKFIT, England (Figure 3) at the chemistry laboratory, University of Lagos. The process entailed that 1.5 liters of n-hexane was poured in a round bottom flask of the soxhlet apparatus. Then, 770 g of crushed and sieved CNSs was wrapped in a filter paper, tied with a clean white thread, and placed inside the soxhlet apparatus thimble. This was followed by heating the n-hexane solvent to 68 ºC in a reflux unit. The cycle of thermal treatment and cooling lasted for 8 hrs until substantial volume of cashew nut shell liquid was achieved. The mixture of n-hexane and cashew nut shell liquid was then fed into a vacuum rotary evaporator to effect the separation of CNSL from n-hexane solvent. The CNSL yield was evaluated using Eqn. 1.

$$\%\,Yield = EL \times 100/Cs \quad (1)$$

where; E_L is volume of CNSL extract;

C_S is mass of cashew nut-shells utilized.

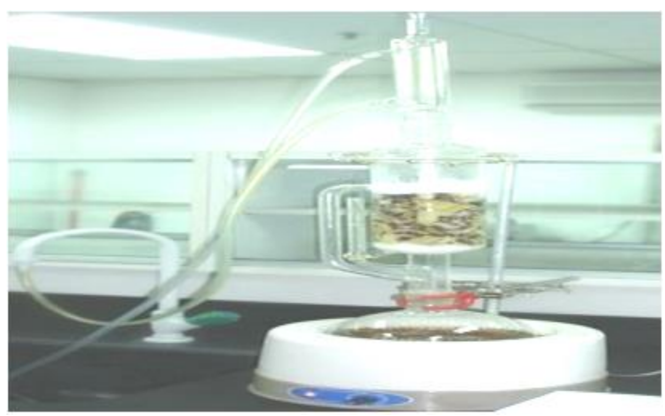

Figure 3: Soxhlet extraction set-up.

2) Characterisation

Physicochemical analysis of CNSL

The analysis focuses on nine (9) critical parameters that influence lubrication potentials of the CNSL. These parameters include density, moisture content, free fatty acid (FFA), viscosity, Saponification value, Iodine value, fiction coefficient, flash-point/fire-point and pH value. The experiment to determine these parameters was carried out at Spectral Laboratory Services and Engineering Analysis, Tundunwada, Kaduna state, Nigeria. The relevant standard procedures employed for each analysis are presented below:

<u>Density</u>

In conformity to ASTM-D4052 standard, determination of the oil density entailed the use of washed and dried 25 ml pyrometer bottle. The washed bottle was filled with water, weighed and then filled with CNSL extract samples alternately and weighed. The oil density was calculated using Eqn. 2.

$$Density = (Wt. of\ 50\ ml\ of\ oil) \div (Vol. of\ 50ml\ of\ oil) \quad (2)$$

<u>Moisture content determination</u>

Half gramme of the oil sample was weighed into a crucible and the weight of sample plus crucible was noted. The sample was kept in an oven at 100 ^oC for 2 hrs, then removed and cooled in a desiccator. Again, the sample plus crucible was weighed and the weight recorded. The moisture content was computed using Eqn. 3.

$$Moisture\ content = (Initial\ oil\ wt. - Final\ oil\ wt) \div (Finaloil\ wt) \times 100 \quad (3)$$

<u>Free Fatty Acid (FFA)</u>

This was evaluated using ASTM-D5559 standard where a mixture of 25 ml petroleum-ether and 25 ml ethanol coupled with 4 drops of phenolphthalein was put inside a conical flask that contained 2 g CNSL. Then, the solution was shaken vigorously for 12 s and titrated using 0.1M KOH till the colour changed to pink. The FFA value was obtained using Eqn. 4.

$$Acid\ value = Titre(ml) \times 0.1 \times 56.10) \div Mass\ of\ oil\ \times 2(g) \quad (4)$$

$$Free\ fatty\ acid = Acid\ value$$

<u>Viscosity</u>

ASTM-D445 standard procedure was followed to test for the oil viscosity. The viscosity value was obtained using a digital viscometer; model, DVI, Brookfield, USA (Figure 4).

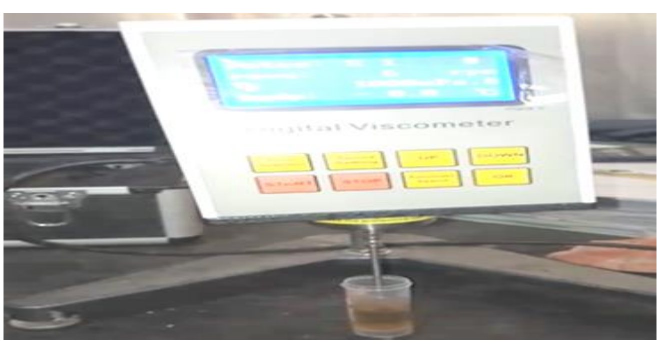

Figure 4: Digital viscosity tester.

<u>Saponification value</u>

According to ASTM-D5558 standard test procedure, the experiment entailed addition of 1 g CNSL sample with 25 ml KOH in a conical flask. This was followed by heating the mixture in an oven for 5 min at 103 ^oC. Then, 1ml phenolphthalein was added to the heated solution and titrated using 0.5M HCl. Eventually, a dry blank was conducted and the observations noted. Eqn. 5 was employed to compute the CNSL Saponification value.

$$\text{Saponification value} = (B - S) \times 28.05 \div (2 \times wt. of\ sample) \quad (5)$$

B is blank titre value = 30.9 cm^3, S is CNSL sample titre value = 16.28 cm^3

<u>Iodine value</u>

The test was carried in conformity to EN ISO 3961 standard. Sample of the oil extract was put inside a dry 250 ml glass-stopper bottle with a small rod added. In line with standard procedure, the highest expected iodine value was divided by 20 to obtain the weight (g) of the oil. Then, 20 ml *Wiji's* solution was added to 10 ml carbon tetrachloride in which a stopper moistened in potassium iodine was inserted and the mixture was hid from light for 25 min. Again, the mixture of 15 ml potassium iodide and 100 ml water was titrated using 0.1M thiosulphate. While this process was being conducted, a blank was carried out simultaneously using 10 ml carbon tetrachloride. Eqn. 6 was used to determine the iodine value.

$$Iodine\ value = (B - S) \times 12.69 \div (0.5wt. of\ sample) \quad (6)$$

B; blank titre value = 49.14 cm^3 and S; oil extract titre value = 5.04 cm^3

<u>Friction coefficient</u>

ASTM-D5183 standard test procedure was followed to determine the friction coefficient of the oil using Eqn. 7 (Huang, 2020).

$$f = 6\pi\eta r v \quad (7)$$

where $\boldsymbol{f}$ is the friction coefficient, $\boldsymbol{v}$ is the velocity of the falling ball with radius **r**. The movement of a spherical ball through a liquid medium (gel) of the viscosity **η,** while $\boldsymbol{\pi}$ is pie (3.142) and 6 is the friction coefficient constant.

<u>Flash Point determination</u>

Flash point temperature was determined according to ASTM-D93 standard with the aid of a heat resistance glass-cup placed on a heating mantle; model EMA 2000/CEB, Bamstead Electro-thermal, UK. The sample was put inside the glass cup, stirred and gradually heated to homogenise the mixture while the temperature was being monitored with the aid of a thermometer. Thus, the temperature at which the oil extract sample's flame increases like a flash was recorded as the flash point. Figure 5 shows the flash point/fire point meter; model BK-FP261, PMCC, China.

Figure 5: Flash point/Fire point meter.

Determination of pH value

Digital pH meter, model 335/5862, Systronics, China (Figure 6) was used to measure the pH of the oil. The test was performed in conformity to ASTM-D6866 standard where 20 ml sample of the oil poured into a beaker in which the pH meter's probe was inserted. The reading on the pH meter was noted after it became stable.

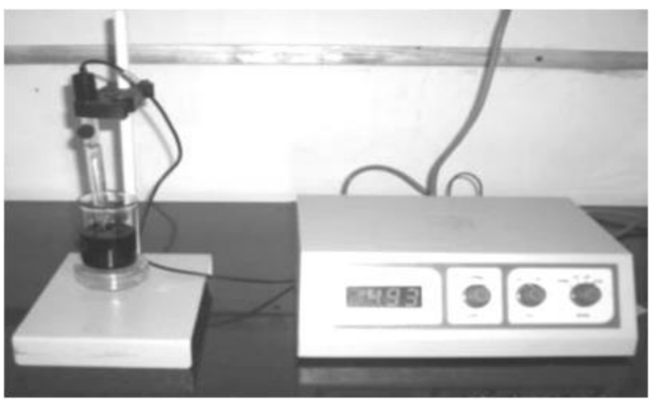

Figure 6: Digital pH meter.

III. RESULTS AND DISCUSSION

A. CNSL Output

The CNSL output through soxhlet extraction process is presented in Table 1. The relatively high yield (45.61%) is attributable to two factors including technology and chemical actions. Himabindu *et al* (2015) posited that soxhlet extraction method is often carried out under controlled conditions. In addition, the chemical used during extraction actually boost the quantity of CNSL extracted within a relatively short time (Efavi *et al*, 2018). Subbarao *et al* (2011), reported achievement of 550 x 10^{-3} m^3 CNSL extracted from 5 kg CNS in 5 min. This is adjudged to be comparable with the current study output of 479 x 10^{-3} m^3 extracted from 770 g CNS in 5 min.

Table 1: CNSL yield

Parameters	Extraction Process
	Soxhlet
Mass of CNS used (g)	770
Mass of CNSL extracted (g)	351.23
Yield (%)	45.61

B. Physicochemical Characteristics of CNSL

Results of the physicochemical analysis on CNSL are made evident in Table 2. The specific gravity at 30 ^{0}C is 0.959 which agrees with literature to the effect that CNSL specific gravity is less than that of water. At ambient temperature, the CNSL density is 0.94 g/cm^3; this is comparable with that of commercial Chevron lubricant oil, 0.99 g/cm^3. The acid value obtained for CNSL is 11.32 mg.KOH/g. This is marginally lower than the range of 12.10 – 15.40 mg KOH/g reported by Balgude and Sabnis (2014). Acid value is usually employed in the measurement of the level of free fatty acids in a given amount of oil. This also depends on the extent of rancidity that is used as an index of freshness (Kyei *et al*, 2019).

According to Sharma *et al* (2015), the acid value demonstrated by CNSL is an indication of its freshness devoid of any traces of degradation. This characteristic anti-rancid behaviour shows that CNSL has a potential application as base oil for synthesis of high quality lubricant and as anticorrosion coating material. Furthermore, the CNSL free fatty acid (FFA) content is 5.66% compared with 6.10 – 7.80% reported by Risfaheri *et al* (2009). Free fatty acid concentration in oil is indicative of its suitability in terms of level of resistance to enzymatic hydrolysis or degeneration (Japir *et al*, 2017). The oil ester value, 127.92 mg.KOH/g is obtained by subtracting the acid value from the saponification value. This relatively substantial ester level can be attributed to a rather paltry presence of free fatty acid of 5.66% CNSL. It was reported that a high ester level indicates the presence of low molecular weight fatty acid (Belsare and Badne, 2017). Thus, the 139.24 mg.KOH/g saponification value is higher than the range of 47-58 mg KOH/g obtained by Balgude and Sabnis (2014). This portends desirable usefulness of CNSL in most lubricant blends owing to the relatively low molecular weight of fatty acids (Japir *et al*, 2017; Wardana *et al*, 2018).

Table 2 shows that CNSL had an average pH of 4.61, that means, the oil is slightly acidic. This phenomenon may be attributed to anacardic acid in technical grade CNSL according to Kanehashi *et al* (2015). Coupled with iodine value of 40.21 gI_2/100g indicating low degree of saturation, CNSL is capable of experiencing moderate reactivity of the double bond as is the case with most non-drying oil.

Table 2: Comparative physicochemical properties of CNSL.

Parameters	Experimental results	Literature (Bagude and Sabnis, 2014; Kyei *et al*, 2019)	Commercial Lubricant (Chevron Soluble oil)
Density (g/cm^3) (@ 30 ^{0}C)	0.94 ± 0.001	0.93 ± 0.03	0.99
Viscosity (@ 100 ^{0}C (Pa.s)	14.12 ± 0.02	*n.a	15.6
Flash Point (^{0}C)	286.63 ± 0.03	n.a	187
pH value	4.61 ± 0.01	5.65 ± 0.03	6.2
Coefficient of Friction	0.001 ± 0.0001	n.a	0.002
Free Fatty Acid (%)	5.66 ± 0.002	4.12 ± 0.4	n.a
Acid Value (mg KOH/g)	11.32 ± 0.001	8.25 ± 0.2	n.a
Saponification value (mg/g)	139.24 ± 0.5	255.26 ± 0.8	n.a
Ester value	127.92 ± 0.01	247.01 ± 0.1	n.a
Iodine Value (gI_2/100g)	40.21 ± 0.01	n.a	n.a
Moisture Content (%)	3.96 ± 0.01	4.45 ± 0.02	n.a

It is observed that CNSL's low moisture content of 3.96% compared positively with values found in literature which indicates the potential durability of the oil given the correlation between the rate of microbial growth and water concentration. The CNSL has its flash point at 286.63 °C compared to 221 °C for the commercial lubricant while its viscosity at 100 °C is 14.12 Pa.s against 15.6 Pa.s of the commercial lubricant (Chevron soluble oil). It is to be noted that the result data presented under result column of Table 2 are the average value for each parameter tested.

IV. CONCLUSION

Extraction of CNSL through soxhlet process was successfully executed. Both the CNSL yield (45.61%) and its critical metal forming lubrication characteristics compared well with established parameters. This was demonstrated by the results of physicochemical analysis carried out on the CNSL with regard to density (0.94 g/cm^3) and moisture content (3.96%). Equally comparable are the values obtained for the critical lubrication parameters namely acid value (11.32 mg.KOH/g), free fatty acid (5.66%), ester value (127.92), saponification value (139.24 mg/g), iodine value (40.21 gI_2/100g), pH value (4.61), friction coefficient (0.001), flash point (286.63 °C) and viscosity (14.12 Pa.s). The major contributions made by this study include among others:

i) Value addition to cashew nut shells waste conversion into a viable metal forming lubrication fluid.
ii) Boost to the quest for a cleaner environment given that substantial tonnage of cashew nut-shells waste disposal challenges is significantly mitigated.

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