Evaluation of Physicochemical and Biodegradability Properties of Selected Nigerian Non-Edible Oilseeds as Potential Cutting Fluids

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Abstract. This paper reports evaluation of physicochemical and biodegradability properties of selected non edible Nigerian oilseeds as a potential cutting fluid. Oil extraction process was carried on the oilseeds, with physicochemical parameters and biodegradability of the extracts were equally assessed. The established physicochemical parameters were percentage oil yield (5.58-61.8%), specific gravity (0.86-0.94), acid value (2.89-18.2 mgKOH/g), iodine value (15.7-104 mg iodine/g), peroxide value (1.35-10.9 mg/g oil), saponification value (173-286 mg KOH/g) and viscosity (37.9-53.1centipoises), while biodegradability ranged between (50.0-63.8%) in comparison with the mineral oil with values less than 20%. Based on this study, the oil extracts of *Caesalpinia bonduc* and *Calophyllum inophyllum* appeared to be the most suitable as potential cutting fluids for further formulation studies and machining trials.

Keywords: cutting fluids, vegetable oilseeds, biodegradability, bacterial inoculums

Introduction

There is an increasing environmental concern on the use of mineral oil-based cutting fluids due to their nonbiodegradability and metal-laden when used. Vegetablebased oil as cutting fluid is already gaining acceptance as alternative to the commonly used mineral oil.

Cutting fluids are one of the types of lubricants, which are extensively used in machining operations. These fluids increase productivity and the quality of manufacturing operations by cooling and lubricating industrial machines during metal forming and cutting processes (Julieb et al., 2003). There are different types of cutting fluids viz mineral oils or mineral based cutting fluids, synthetic and semi-synthetic cutting fluids. Mineral oils are petroleum based oil and are the most commonly used cutting fluid (Lawal et al., 2012). Their properties depend on the chain length, structure and refining level of the petroleum. Mineral oil has poor biodegradability with a notable shortcoming of inducing long term pollution of the environment. Mineral based oil is also a limited and steadily decreasing resource whereas, the vegetable based oils are sustainable.

Oilseeds on the other hand are biodegradable, renewable, non-toxic, and affordable; possess good lubricity and high viscosity index when compared with mineral oils (Krahenbuhl, 2002). Vegetable oils primarily consist of triglycerides, which are glycerol molecules with distinguished fatty acid chain structures (Fox and

As mineral oil is non-biodegradable, toxic and nonrenewable, there are problems with its disposal. This is the major reason why vegetable oilseeds extracts are given consideration as an alternative to the commonly used mineral oils in the lubricant industries and several other industrial applications. Poor disposal of cutting fluids and management when spent, may result in enormous health hazards (Sokovic and Mijanovic, 2001). Some addictives used in mineral oil cutting fluids formulation are considered to be hazardous to the environment and human health (Shokrani et al., 2012). Other notable limitations of mineral oils that have been identified, in comparison with its vegetable based counterparts are less surface adhering ability (Krahenbuhl, 2002; Woods, 2005), weaker layer of metal surface lubrication (Krahenbuhl, 2002), lower viscosity index, lower flash point, lower boiling point and molecular weight (Khan and Dhar, 2006).

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Stachowiak, 2007). This confers special advantages on lubrication. It is due to these long and polar fatty acid chains, which provide high strength lubricant films that interact strongly with metallic surfaces, reducing both friction and wear (Matthew *et al.*, 2007). The polarity of fatty acids produces oriented molecular films, which provides oiliness and imparts anti wear properties. Fatty acids are thus believed to be key substances with regard to lubricity (Maleque *et al.*, 2003).

Good oilseed extracts that will be most suitable for replacement and trial as cutting fluid assessment must combine the following characteristics appreciable oil yield quality, good viscosity range, resistant to air oxidation due to possession of high level of saturated fatty acids (Fox and Stachowiak, 2007; Jayadas and Nair, 2006), and easy microbial degradation in the environment (Erhan and Asadaukas, 2000).

These attributes of vegetable based oilseeds would enhance their usefulness as bio fuels, cutting fluids, transformer oil and industrial lubricants (Honary, 2004).

However, disadvantages of vegetable oils as a cutting fluid include: perceived poor hydrolytic stability and limited thermal and oxidation stability (Abdalla and Patel, 2006). These shortcomings of vegetable oils for lubrication are corrected for the use of addictives for optimal lubrication performance.

Nigeria is endowed with a lot of edible and non-edible oilseeds. Since there are lots of pressure on edible oilseeds, non-edible oilseeds that exist mostly unused can be considered for use as cutting fluids. Therefore, this study is aimed at assessing some non edible Nigerian oilseed plants as potential cutting fluids. This was done by carrying out detailed physicochemical tests (specific gravity, viscosity, acid value, iodine value, peroxide value and saponification value) on the oilseeds extracts and a biodegradability test effected by using two strains of bacterial inoculum, *Pseudomonas aeruginosa* (P a) and *Bacillus subtilis* (B s), to feed on carbon sources provided by the oil extracts on an incubated medium.

Materials and Methods

Sample description and preparation. Seeds samples collected for the study were *Citrullus lanatus* (watermelon), *Chrysophyllum albidum* (star apple), *Calophyllum inophyllum, Hura crepitans* (sand box), *Caesalpinia bonduc* (nicker nut), *Jatropha curcas* (barbados nut) and *Magnifera indica* (mango). Table 1 shows the english, botanical and local names of the oilseeds used for this study. Seeds were carefully picked around Ibadan metropolis, sun dried and unshelled. About 1.3 kg of the unshelled seeds was crushed and ground using a stainless steel blender for repeated oil extraction processes. The oil extraction process was carried out with the use of a Soxhlet extractor and *n*-hexane was used as the extracting solvent.

Soxhlet extraction of oil seeds. Oil samples were extracted using a 500 mL round bottom flask, Soxhlet apparatus and *n*-hexane of boiling range between 40-60 °C according to the (AOAC, 1975) procedure.

Physicochemical characterisation of oil extracts. Physicochemical characterisation of oil extracts was performed using different AOAC methods.

Acid value of oil extracts. The acid value was determined using the AOAC (1984) method and acid value calculations effected using the below equation.

Acid value =
$$\frac{56.1 \times M \times V}{W}$$
.....(1)

where:

M = molarity of KOH (0.1 M); V = volume of KOH (mL); W = weight of sample (g).

Saponification value. The saponification value was evaluated using the AOAC (1984) and the calculation carried out using the equation:

where:

 V_0 = the volume of the solution for blank (mL); V_1 = the volume of the sample solution (mL); M = molarity of the HCl (M); W = weight of the sample (g).

 Table 1. English, botanical and local names of the samples

English name	Botanical name	Local name
Watermelon	Citrullus lanatus	NA
Barbados nut	Jatropha curcas	Eso Lapalapa
Sand box	Hura crepitans	Atapara
NA	Calophyllum inophyllum	NA
Mango	Magnifera indica	Mangoro
Nicker nut	Caesalpinia bonduc	Eso Ayo
Star apple	Chrysophyllum albidum	Agbalumo

NA = not available.

Iodine value =
$$\frac{(B-S) \times M \times 0.1269 \times 100}{W} \dots (3)$$

where:

B = blank titre volume (mL); S = sample titre volume (mL); M = molarity of sodium thiosulphate used (M); W = weight of oil sample (g).

Peroxide value. The peroxide value was evaluated using the AOAC (1984) and the calculation effected by using the equation:

$$PV = [1000 (v_1 - v_2) \times M] / W \dots (4)$$

where:

W = weight of sample (1 g); v_1 = sample titre volume of thiosulphate (mL); v_2 = blank titre volume of thiosulphate (mL); M = molarity of thiosulphate (M).

Specific gravity (at 25 °C). The specific gravity bottle was used to determine the specific gravity of the extracted oil by comparing the weight of oil to the weight of an equal volume of distilled water.

Viscosity (1) (at 25 °C). The viscosity of the oil was measured using Oswald kinematic viscometer. The extracted oil, viscometer, 20% sucrose solution and water were conditioned at a temperature of 25 °C in water bath. Water volume of about 9 cm³ was poured into the viscometer and drawn above the upper mark using suction. As the water drops in the tube, the time at which the meniscus passes the lower mark was measured with a stopwatch. Then oil sample added into the viscometer, the time for the passage of the oil between meniscuses was determined.

Viscosity value (in centipoises) was determined using the equations (5) and (6):

In the Oswald viscometer, the viscosity of a liquid is given in terms of time of flow between two marks by the equation:

where:

d = density in g/mL; η = viscosity in centipoises; t = time in seconds.

A and B depends on the design of the instrument, and are determined by calibration with two liquids of known viscosity (I) and density (d). Here, water (d = 0.9971 g/mL, I) = 0.891 centipoises and sucrose (d = 1.0794 g/mL, I) = 1.699 centipoises). The average value of t recorded for both water and sucrose together with their respective viscosity and density were inserted into equation (5) above, after which the values of A and B were calculated out. The average time t, taken for the oils were inserted into the equation (6), and their viscosities obtained.

Determination of biodegradability of oil extracts. Quality assurance of the biodegradability test was carried out by sterilising all glassware in hot air oven before use. The working bench was also surface sterilised with 70% ethanol. The bacterial species used for the study (test isolates) were obtained from the Department of Microbiology, University of Ibadan, Ibadan, Nigeria. The test isolates were sub-cultured into nutrient agar plates and incubated at 37 °C for 24 h. Isolates used were *Pseudomonas aeruginosa* (P a) and *Bacillus subtilis* (B s).

Preparation of the media. The method of Bushnell and Hass (1941) was used to prepare the media. The media used were prepared following the instructions of the manufacturer: 0.02 g of MgSO₄, 0.02 g CaCl₂, 0.1 g of KH₂PO₄, 0.10 g of K₂HPO₄, 0.10 g of NH₃NO₃, 0.005 g of FeCl₃ and 3.0 g of yeast extract, all dispensed in 100 mL distilled water and the medium was maintained at pH 7.0 using appropriate acid or alkali (NaOH and HCl). The solutions were later sterilised at 121 °C for 15 min inside an autoclave and allowed to cool to room temperature.

Preparation of inoculums and the biodegradability test. Shake flask test (US EPA, 1998) was used to prepare the inoculums for the biodegradability test. The bacterial cultures (24 h old) from enrichment medium were inoculated in the above selected mineral salts medium in a 100 mL capacity conical flask, with 0.5 g oil seeds extracts in each conical flask as the sole carbon source for the medium.

The content of each conical flask were kept in a shaker at 190 rpm and 30 °C for 120 h.

Each shaker contained 25 mL of sterile selective mineral salts medium with 0.5 g of the oil extract and the bacterial culture of *P. aeruginosa* (P a) and *B. subtilis* (B s) and a control containing neither of the bacterial strains.

After 5 days of action by the bacterial strains on the medium, the conical flasks containing the medium were removed from the shaker, centrifuged using a high speed refrigerated centrifuge at 4000 revolution per minute for 15 min. Total organic carbon (TOC) of contents of the different conical flasks that were centrifuged, were analysed. Comparisons were made between the TOC values of the oil seeds controls and those that were degraded using different bacterial strains. Biodegrad-ability was calculated by finding the percentage of the TOC of the control medium that was degraded by the media containing the bacterial cultures.

Determination of total organic carbon. Total organic matter (TOC) contents of the control and bacterial degraded oil extracts were estimated by oxidation of organic carbon (Ademoroti, 1996). The procedure involved oxidation of TOC in the controls and unused organic carbon in the degraded samples by potassium dichromate which is a strong oxidising agent in the presence of concentrated acid.

Bacterial degraded oil extract medium (1.0 g of the centrifuged) was measured into a 250 mL conical flask. 10 mL of 0.5 M $K_2Cr_2O_7$ was added and gently stirred. 20 mL of concentrated H_2SO_4 was rapidly added and carefully swirled to avoid splashing, until all the reagents were mixed. The flask was allowed to stand for 20 min and 100 mL of distilled water was added afterward.

The solution was cooled and titrated against standardised 0.25 M ferrous ammonium sulphate (FAS) solution to a wine red colour at the end, using 3 drops of ferroin indicator. The FAS titre value was recorded as Vs (mL). A blank determination using the above procedure was done without the sample. The FAS titre value for the blank titration was recorded as V_b (mL).

% TOC =
$$\frac{(V_b - V_s) \times M \times K}{\text{weight of sample (g)}}$$
.....(7)

where:

 V_b = FAS titre value for blank titration; V_s = FAS titre value for sample titration; M = molarity of standardised FAS; K = 1.38.

The percent biodegradability was calculated from the results obtained for TOC of the control container and the value obtained using two different strains of the bacterial inoculums. The biodegradability was determined as:

Biodegradability=
$$\frac{(\text{TOC}_{\text{control}} - \text{TOC}_{\text{bacterial inoculums}})}{\text{TOC}_{\text{control}}} \times 100...(8)$$

Results and Discussion

Oil seeds extract characterisation. The physicochemical properties of the oil extracts from the oil seeds are shown in Table 2.

Table 2. Physicochemical properties of Nigerian non edible oilseed samples

Oilseeds	State at RT	Colour	Oil yield (%)	Specific gravity	Acid value (mgKOH/g)	Iodine value (mg iodine/g)	Peroxide value (mg/g oil)	Saponification value (mg KOH/g)	Viscosity (cP)
Citrullus lanatus	Liquid	Light yellow	40.5	0.90	3.24±0.04	104±0.40	10.9±0.17	185±0.46	43.3
Jatropha curcas	Liquid	Light yellow	46.4	0.89	5.28±0.32	98.8±0.54	1.35±0.05	173±0.42	40.5
Hura crepitans	Liquid	Light yellow	35.7	0.93	18.2±0.55	15.7±0.38	2.30±0.14	286±0.36	37.9
Calophyllum inophyllum	Liquid	Greenish yellow	61.8	0.94	33.9±0.21	62.5±0.58	2.05±0.06	196±0.48	53.1
Magnifera indica	Semi solid soft fat	Pale yellow	10.3	0.91	2.89±0.04	45.9±0.36	3.10±0.42	180±1.49	NA
Caesalpinia bonduc	Liquid	Golden brown	33.7	0.90	3.17±0.04	25.1±0.36	2.10±0.16	257±1.99	47.9
Chrysophyllum albidum	Liquid	Dark yellow	5.58	0.86	3.59±0.08	53.6±0.35	1.70±0.15	201±2.40	NA

RT = room temperature; NA = not available; cP = centipoises.

The colour of the seed extracts varied from different shades of yellow to dark brown. The state at room temperature was generally liquid, except for mango seed extract, which was observed as semi solid soft fat, a similar observation was reported by Saiprabha and Goswami-Giri (2011).

The density of oil relative to that of an equal volume of water (specific gravity) ranged from 0.86 to 0.94. These values are within the range of specific gravities reported for related oilseeds extract from some selected tropical seeds (Sabinus and Oscar, 2012).

A previous report by Ekpa and Ekpe (1995) showed that, unlike free fatty acid content, which is a measure of free fatty acid present in a fat or oil, acid value is a measure of total acidity of the lipid, involving contributions from all the constituent fatty acids that make up the glyceride molecule. As oil-fats become rancid, triglycerides are converted into fatty acids and glycerol and causing an increase in acid values. Besides, better information on the acidity of glycerides could be obtained from the acid value, which takes into account the contribution of all the constituent fatty acids in the oil or fat. The total acidity, expressed as acid value, was highest in C. inophyllum oil, with 33.9 (mgKOH/g), followed by H. crepitans (18.2 mgKOH/g) and was lowest in M. indica (2.89 mgKOH/g) (Babalola and Apata, 2011). It supported that low acid value is one of the most important yardsticks for measurement and assessment of edibility of vegetable oils and high values, a pointer to the tendency for enormous industrial applications of the concerned vegetable oil. Hence, oil extracts with higher acid values will serve better as cutting fluids.

Saponification value measures the average size of fatty acid present in an oil sample which depends upon the molecular weight and percentage concentration of fatty acids components in it. Saponification value will be higher if the oil contains more of saturated fatty acids (C14:0, C16:0, C18:0) as it determines the length of carbon chain present in the oil (Muhammad *et al.*, 2011).

Saponification values varied significantly among the oils and was highest in *H. crepitans* oil (286 mg KOH/g), followed by *C. bonduc* oil (257 mg KOH/g) while, the lowest value was in *Jatropha curcas* oil (173 mg KOH/g). Since there is an inverse relationship between saponification value and weight of fatty acids in the oil, it can be inferred as reported by Dosunmu and Ochu (1995) that the oil extracts with high saponification values above 190 mg KOH/g, have greater number of fatty

acids of low molecular weight, which is a preferred range for vegetable-based cutting fluids (Shashidhara and Jayaram, 2010). This is understandable because of the relative ease of oxidation of high molecular weight fatty acid. Also low molecular weight fatty acids are soluble in water during cutting fluid formulation.

The usual method of assessment of hydro peroxides (primary oxidation products) is by determination of peroxide value (Gunstone, 2004). C. lanatus (watermelon) has a very high peroxide value (10.9 mg/g oil) hence, it will be less stable and would easily undergo deterioration when exposed to atmospheric oxygen, a condition which operates during the use of cutting fluid in the course of machining. Moreover, low peroxide value suggests that the oils are fresh and could be stored for a long period of time without getting rancid. J. curcas oil, C. albidum, C. bonduc, C. inophyllum oils and H. crepitans with peroxide values of: 1.35, 1.70, 2.10, 2.05 and 2.30 mg/g oil, respectively, all showed low peroxide values, thus, proving the oxidative stabilities required of the seed oil with significant cutting fluid properties.

Vegetable oils containing a large percentage of monounsaturated fatty acids will be the most likely candidates for vegetable-based lubricants. This is because they have greater oxidative stability than polyunsaturated oils, and also will remain as a fluid over a much larger range of temperatures.

The iodine value is a measure of the unsaturation of fats and oils. Higher iodine value indicates higher unsaturation of fats and oils (Knothe, 2002). This value can be used to infer the extent of double bonds present in the oil i.e. unsaturation, which reflects the susceptibility of oil to oxidation. The iodine value obtained for the extracts of C. lanatus and J. curcas, with iodine values of 104 and 98.8 mg/g iodine, respectively, were placed in the semi drying oil range. H. crepitans oil, C. bonduc and M. indica oil, with iodine values of 15.7, 25.1 and 45.9 mg/g iodine, respectively, are believed to be made up of predominantly saturated and monounsaturated fatty acids and thus most suitable for use as cutting fluids where, high level of oil saturation is required (Fox and Stachowiak, 2007; Jayadas and Nair, 2006). C. albidum and C. inophyllum with iodine values of 53.6 and 62.5 mg/g iodine, respectively, also meet up with cutting fluid requirements based on their iodine values.

Little or no variation, perhaps, due to climatic conditions was observed between the values of physicochemical properties of oil seed obtained in this study and those reported for other purposes (Osamudiamen and Afolabi, 2012; Ochigbo and Paiko, 2011; Saiprabha and Goswami-Giri, 2011; Ajayi, 2010; Olatidoye *et al.*, 2010; Oderinde *et al.*, 2009; Emil *et al.*, 2009; Taiwo *et al.*, 2008). Reasons for this little variability in results may have resulted from similarities in the species of the oilseeds used and the extracting solvents.

Petroleum/mineral lubricating oils used as cutting fluids are generally high-viscosity base stocks, with viscosity in the range of 15 to 50 cSt at 100 °F (Salete and Joa[°]o Fernando, 2006). High-viscosity oils accelerate heat removal from the tool and work piece because of better heat transfer coefficients, and they facilitate the rapid settling of chips and swarfs from circulating oil. The viscosity values obtained for the oil extracts (*C. bonduc* 47.9 cP, *C. lanatus* 43.3 cP, *J. curcas* 40.5 cP, *H. crepitans* 37.9 cP, *C. inophyllum* 53.1 cP) falls within the preferred range of oil that could function or perform as a cutting fluid.

Vegetable oils have also been applied as transformer coolant/lubricant oils and have been found to conform to all industry standards with performances and cost profiles comparable to the conventional mineral oils applied in transformer cooling (Abb Inc. 2002; Masjuki et al., 1999). Transformer oil products have been produced from soybean oils as well as castor oils. Castor oil has been used as industrial lubricant and hydraulic brake fluids for enormous industrial machining (Marter, 1981). Physicochemical characterisation of crude castor oil has been reported by Akpan et al. (2006). Specific gravity of 0.9587, acid value of 1.148 (mg KOH/g), saponification value of 185 (mgKOH/g) and iodine value of 87.72 mg/g iodine were documented for castor oil. Comparing these physicochemical parameters of castor oil with the seeds oil under test, with much emphasis on the iodine value range, the oil extracts of C. inophyllum, C. albidum, M. indica, C. bonduc and H. crepitans possess potentials to be used as cutting fluid.

Moreover, results of *M. indica* (oil yield = 10.3%, S.G = 0.91, saponification value = 180 mg KOH/g, peroxide value = 3.10 mg/g, acid value = 2.89 mg KOH/g) compares with the values obtained previously (Saiprabha and Goswami-Giri, 2011), for *M. indica*; *C. albidum*, (oil yield = 5.58%, S.G = 0.86, acid value = 3.59 mg KOH/g, iodine value = 53.6 mg/g, saponification value = 201 mg KOH/g, peroxide value = 1.70 mg/g) varies slightly with values reported for the same seed by

Ochigbo and Paiko (2011) due to difference in the extracting solvents. While they used a mixture of solvents (hexane and petroleum ether), only *n*-hexane was used in this study. However, the observed values from this study were in accordance with the values reported by Osamudiamen and Afolabi (2012).

Biodegradability of the tested oilseeds. Table 3 summarises the results of the biodegradability test of the oil extracts by the two species of bacterial inoculums: *Pseudomonas aeruginosa* (P a) and *Bacillus subtilis* (B s). (P a) degraded *C. lanatus* with 5.09%, which is a much smaller value when compared with *B. subtilis* (B s) with 55.9%. *C. inophyllum* and *J. curcas* oil extracts were 57.0 and 58.1%, respectively, degraded by (P a). 7.48 and 4.63% by (B s) was only effective in degrading *C. bonduc* by 63.8% and *H. crepitans* 23.1% better than (P a) with 9.50 and 16.6%, respectively. The two strains of bacterial inoculums used did not show any clear preference on the degradation of the mineral oil used in the biodegradation test carried out (P a) of 15.0% and (B s) with 18.1%.

Percentage biodegradability of the oil extracts ranged between (50.0-63.8%) effected by either strain of the bacterial species on different oilseeds extracts, which is not small value comparable to the value recorded with the mineral oil used of (P a) 15.0% and (B s) with 18.1%.

Table 3. TOC of control and bacterial inoculums and% biodegradability

Oilseeds	TOC (control)	TOC (P a)	TOC (B s)	Biodegradability (%)
Citrullus lanatus	3.908	3.709	1.722	P a = 5.09 B s = 55.9
Caesalpinia bonduc	7.319	6.624	2.650	P a = 9.50 B s = 63.8
Magnifera indica	4.902	3.974	4.471	P a = 18.9 B s = 8.79
Hura crepitans	2.583	2.153	1.987	P a = 16.6 B s = 23.1
Calophyllum inophyllum	3.544	1.524	3.279	P a = 57.0 B s = 7.48
Jatropha curcas	2.848	1.192	2.716	P a = 58.1 B s = 4.63
Mineral oil	0.6624	0.5630	0.5420	P a = 15.0 B s = 18.1

TOC = total organic carbon; P a = *Pseudomonas aeruginosa*; B s = *Bacillus subtilis*

Biodegradability of vegetable oils is the strongest point in their consideration for use as a potential cutting fluid. In the light of more concerns about the environmental impact of the use of industrial fuels and lubricants, they offer in theory the most plausible solution to the issue of obtaining renewable and eco-friendly lubricants and fuels.

Conclusion

In the screening exercise conducted in this study, Caesalpinia bonduc with oil yield of 33.7%, iodine value of 25.1 mg/g oil (depicting high level of saturated fatty acids composition), peroxide value of 2.10 mg/g oil (showing resistance to peroxidation), viscosity of 47.9 centipoises and a good biodegradability level of 63.8% on Bacillus subtilis bacterial species, closely followed by Calophyllum inophyllum with percent oil vield of 61.8%, iodine value of 62.5 mg/g of iodine, peroxide value of 2.05 mg/g oil, viscosity of 53.1 centipoises and a good biodegradability of 57.0% effected by Pseudomonas auerogenosa bacterial specie, are the most suitable oil extracts for trial as cutting fluids. These seeds are readily available in Nigeria, most especially in the south-western part of the country with no significant usage at present. Hence, there exists no noticeable challenge as regards availability when considered for use as cutting fluids. Other seeds with good promise but little shortcomings, by virtue of their physicochemical parameters, could be structurally and chemically modified before their trial as a cutting fluid.

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