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Physicochemical characteristic of Exudate of *Dacryodes edulis*

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ABSTRACT: The Present work deals with the study of exudate of *Dacryodes edulis* with regards to various seasonal physicochemical properties of the purified solids exudate, acid hydrolysis product and saponification product like charring temperature (^oC) (195.73 ± 4.75, 190 ± 7.9, 190 ± 3.4,); flash point (^oC) (105 ± 5.0, 100 ± 7.9, 100 ± 3.4); moisture content % (1.32 – 1.35, 1.05 – 1.08, 1.00 - 1.10); ash content (%) (1.77 – 1.85, 1.21 – 1.31, 1.26 -1.37); lignin content (%) (7.77 – 7.85, 6.21 – 6.31, 5.26 – 5.37); electrical conductivity (µS/cm) (28.40 -29.20, 32.10 – 33.00, 30.30 – 30.90); density (g/cm^3) (0.76 -0.77, 0.94 – 0.98, 0.88 – 0.98); melting point (^oC) (61.08 – 73.35, 58.69 – 73.35, 55.35 – 65.67). The physicochemical characteristic of *Dacryodes edulis* is found to be fluctuated with seasonal variations during the present investigation. The correlation coefficient showed positive relationship among the properties. The study reveals that exudate of *Daryodes edulis* can be tapped in large quantity in the dry season and is rich in lignin content but the seasons does not affect the properties. © JASEM

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KEY WORDS: *Dacryodes edulis*, Seasonal, Physicochemical properties, purified exudate

Introduction

Exudate are possibly one of the earth's most abundant normal property which gives fuel as well as bonding agent in many parts of South Eastern Nigeria where the plant is mostly originated. It is a watery substance produced on its own accord at the place where the plant is wounded. The quantity increases as the weather become warmer and less when it is cool (Anneline *et. al.*, 2012).

The preservation of exudate as renewable normal property is very much essential. The most important step for preservation of exudate is to maintain a proper treatment with acid or alkalis to modify its gummy nature so that its sticky properties are withdrawn and mucilaginous properties are introduce (Britt,1990). The treatment is directly related to the dispersing capacity in water and other solvents (Blunt 1969).

So, an appropriate administration as paper sizing agent is very much necessary. Some of the research works on *Dacryodes edulis* was those of Akpabio 2014, Udo *et al.2012*, Ayatse and Loike, 1992, Casey,1980, Davin *et al. 1992*, Ekpe *et al. 1976* and Coppen, 2013.

The purpose of the present study is to observe the acid and alkalis treatment of *Dacryodes edulis* by physicochemical procedures and to determine the changes in treatment properties by seasons (dry and wet) and to find the relationship between different physicochemical properties.

MATERIAL AND METHODS

Study area: Akwa Ibom is a state in South-Southern Nigeria which borders on the east by Cross River State, on the west by Rivers State and Abia State, and on the south by the Atlantic Ocean and the southernmost tip of Cross River state. It is comprises of three Senatorial Districts namely. Ikot Ekpene, Uyo and Eket. Climate is divided into two seasons, dry (November –March), rainy (April – October).

The present study was conducted on the physicochemical properties of exudate of *Dacryodes edulis* from Akwa Ibom North – East: Uyo Senatorial District (USD), Akwa Ibom South: Eket Senatorial District (ESD) and Akwa Ibom North west: Ikot Ekpene Senatorial District (IKSD). The Akwa Ibom is located in the coastal Southern part of Nigeria, lying between Latitude 4^{o}32N and 5^{o}33N

*Sampling Methods***:** Sampling (crude exudate from African pear tree) *Dacryodes edulis* was collected randomly from five pear tree at three senatorial Districts of Akwa Ibom State, Nigeria. Many incisions were made on the bark of the Trees and after some few minutes the exudate oozed out of the tree. The fresh exudate was a milky, highly viscous liquid and this was collected with the help of a knife blade and dropped into a container.
Several physicochemical properties such as, charring temperature (^oC), melting point (^oC), moisture content (%), flash point (^oC), Conductivity (µS/cm), ash content (%) density (g/cm^3) and viscosity (poise) has been analyzed.

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Analysis of physicochemical parameters: charring temperature and melting point were measured *in situ* by using melting point apparatus, Moisture, ash, lignin contents and density were determined using standard procedures of the Association of Official Analytical Chemist (AOAC 2002). Electrical conductivity was measured by conductivity meter (Esico Microprocessor based conductivity meter, Model 1601). A u-tube Ostwald viscometer (BS 188) was used for determination of viscosity.

Statistical analysis: *Statistical analysis:* The correlation between various physicochemical properties of exudate samples were analyzed statistically conducting Pearson correlation analysis with the help of SPSS software (16.0).

RESULT AND DISCUSSION

Physicochemical properties (Mean±S.D) of exudate of *Dacryodes edulis* obtained during the present investigation (during November 2012 – October 2014) are presented in table-1

Seasonal Variation of Physicochemical Properties of the Exudate of *Dacryodes edulis*

Table 1: Direct extract Seasonal Variation of Physicochemical Properties

Direct Extract sample	First year study period (November 2012 - October 2013)		Second year study period (November 2013 - October 2014)	
Property	Range	Mean ± SD	Range	Mean ± SD
Charring temperature (^oC)	185 – 210	195.73 ± 4.75	180 - 196	195.53 ± 4.91
Flash point (^oC)	100 – 120	105.33 ± 5.01	99.4 - 105.5	110.00 ± 7.92
Moisture content (%)	1.32 -1.36	1.20± 0.15	1.32 – 1.35	1.23±0.12
Ash content (%)	1.79 – 1.87	1.68± 0.14	1.77 – 1.85	1.54±0.11
Lignin content (%)	7.79 - 7.89	6,84± 1.00	6.77 – 7.85	6.78 ± 0.05
Electrical conductivity (μS/cm)	28.40 – 33.20	30.05 ± 1.59	28.40 -30.20	29.80± 1.62
Density (g/cm^3)	0.76 – 0.98	0.88± 0.21	0.76 - 0.88	0.82 ± 0.02
Melting point (^oC)	61.08 – 73. 35	65.32±5.23	61.08 – 73.35	68.22±3.2
Volume (ml/min.)	8 -100	69.34± 2.3	8.80 – 110	72±1.2

Charring temperature: Charring temperature is one of the essential properties; since it removes oxygen and hydrogen from the exudate, the remaining char is compose primarily of carbon. The ignition and combustion of exudate is mainly based on thermal decomposition of cellulose which determines the amount of gaseous fuel generated by the exudate for the flame. Oxygen status of any charring is greatly influenced by temperature (Wetzel, 1996). Charring temperature recorded as minimum value of 180^{o}C and maximum of 210^{o}C during the two year study period (November 2012 to October 2014) with a mean value of 195.53^{o}C ±4.91 table 4.10. The average charring temperature observed during the first year study period was 195.73^{o}C±4.75 whereas during the second year of observation the mean charring temperature was 195.53^{o}C±4.91. In the present investigation, minimum charring temperature was obtained during wet season and maximum during dry. This investigation is also in close conformity with the finding of Kannan and Job, 1979; Chaturbhuj *et al.* 2004, Mishra *et al.*2008 and Arya *et al.*2011. Charring temperature shows a significant correlation effect between dry and wet season ($p<0.05$ level) with ($r=0.830$).

Flash point: The temperature which exudate gives off enough flammable vapour to form a mixture with air that can be ignited by contact with hot surface, spark or flame is one of the essential properties; It is the lowest temperature at which vapour of exudate will ignite. The vapour may cease to burn when the ignition source is removed but the lower the flash point the greater the heating hazard. Flash point recorded as minimum value of 99.4^{o}C and maximum of 120^{o}C during the two year study period (November 2012 to October 2014) with a mean value of 105.33^{o}C ±5.01 table 4.40. The average flash point observed during the first year study period was 105.33^{o}C±4.75 whereas during the second year of observation the mean charring temperature was 110.93^{o}C±7.92. In the present investigation, minimum flash point was obtained during wet season and maximum during dry season. This investigation is also in close conformity with the finding of Udo *et al.* 2012, flash point shows no significant correlation between the dry and wet season ($p<0.05$ level) with ($r=1.000$) for paired samples.

Moisture content: This is the amount of water quantity contained in the exudate. In the first year of investigation, moisture content revealed a close range of variation with a minimum of 1.32% and maximum of 1.36%. Whereas during the second year of observation the range obtained was 1.32 % to 135%. The moisture content of exudate during the study period shows a significant correlation between the dry and wet season ($p<0.05$) with paired samples ($r= 0.982$) for USD dry and USD wet, IKSD dry and IKSD wet, and ESD dry and ESD wet.

Ash content: Ash content is imparted by the burned off organic matter present in a substance. It serves as the indicator of the inorganic salt (minerals) that were present in the original plant material and is usually compose of calcium, potassium, magnesium; and anions such as carbonate sulphate and silicate. Ash content obtained was in the range of 1.79% to 1.87%

and 1.77% to 1.85% in the first and second year of study period respectively. The highest value shows during the dry season and lowest in the wet season. Similar trend was reported by Udo *et. al*., 2012. It is seen that Ash content shows significant correlation ($p<0.05$) with paired samples (r = 0. 982).

*Lignin content***:** Lignin content is an organic substance binding the cells, fibres and vessels which constitute wood and the lignified elements of plant. It is the most abundant renewable carbon source of the earth. In the present study period i.e. November 2012 – October 2014, lignin content showed a Significant correlation ($p<0.05$) with paired sample between USD dry and USD wet (r = 1.00). It recorded as minimum value of 6.77% and maximum of 7.89% during the two year study period with a mean value of 6.84^oC ±1.00 and 6.78 ± 1.00 tables 1.

Electrical conductivity **(EC):** Electrical conductivity is a numerical expression of the ability of a water sample to carry an electric current. EC values of *Dacrydoes edulis* exudate were in the range of 28.40 to 33.20 (µ S/cm) during the present study (November 2012 – October 2014 showing mean value of 30.05 µS/cm and 29.80±1.02 for the two year study period respectively (table 4.10). Seasonal variations of the present investigations revealed that EC was high during dry seasons and low during wet seasons. This result is supported by the findings of Udeala and Chukwu, 1985, Vain *et al.* 2009 and Usher, 1996. In the present study EC showed significant correlation ($p<0.05$) between the 134paired sample ESD dry and wet (r=1.00), IKSD dry and IKSD wet (r=0.998) and with USD dry and USD wet (r=0.990).

*Volume***:** This is the quantity of exudate obtained from the three senatorial districts. The rates of flow per minutes were observed. The result shows that as the time increases the flow rate also increases. The greatest quantity was obtained from ESD during the dry season and least from IKSD during the wet season. Same observation was recorded during the two years study for November 2012 to October 2013 and November 2013 and October 2014. This result is supported by the findings of Udeala and Chukwu, 1985, Vain *et al.* 2009 and Usher, 1996.

Physicochemical Properties of the Exudate of *Dacryodes edulis* Saponification and Hydrolysis Product

Table 2: Physicochemical Properties of the Exudate of *Dacryodes edulis* compared with Gum Arabic

Property	Gum Arabic	Crude Exudate	Direct extract	Chromatography column	Saponification product	Hydrolysis product
Charring temperature (^oC)	ND	200 ± 3.4	195.73 ± 4.75	200 ± 3.4	190 ± 3.4	190 ± 7.9
Flash point (^oC)	90	110 ± 3.4	105 ± 5.0	110 ± 7.9	100 ± 3.4	100 ± 7.9
Moisture content (%)	9.0 ± 0.58	1.32 – 1.36	1.32 – 1.35	1.32 – 1.34	1.00 1.10	1.05 – 1.08
Ash content (%)	2.94	1.79 – 1.89	1.77 – 1.85	1.87 -1.97	1.26 -1.37	1.21 – 1.31
Lignin content (%)	10.0	7.79 -7.89	7.77 – 7.85	6.87 – 6.97	5.26 – 5.37	6.21 – 6.31
Electrical conductivity (µS/cm)	40.20	ND	28.40 -29.20	28.40 – 28.70	30.30 – 30.90	32.10 – 33.00
Density (g/cm^3)	0.98	ND	0.76 -0.77	0.82 - 0.88	0.88 – 0.98	0.94 – 0.98
Melting point (^oC)	70.50	ND	61.08 – 73.35	ND	55.35 – 65.67	58.69 – 73.35

ND = Not determined

Lignin Content: Lignin is a component of exudate and is 7.79 – 7.89% in both crude exudate and direct purified extract for dry season and reduce to 7.77 – 7. 83 in the wet season, 6.93 – 6.97% in column extract for dry season and 6.87 – 6.91% for wet season, 5.32 – 5.37% for saponification product in the form of soda-lignin for dry season and 5.26 – 5.30% for wet season and 6.27 – 6.31% in acid hydrolysis product as lignosulphurnic acid for dry season and 6.21 – 6.25% for wet season which is less than that of Gum Arabic (10.00 – 15.05%). More lignin is present in the exudate than in the saponification and acid hydrolyzed exudate products, because soda lignin and lignosulphurnic acid are slightly soluble in the liquors.

*Ash Content***:** The amount of ash in crude exudate, according to Casey (1980) lies between 1-3%, studies however, revealed that the major components of wood ash are calcium, potassium, magnesium, and silicate (Masterson, 1988). The ash content in the crude exudate and direct solvent extracted one is 1.80 -1.89 for dry season and 1.77 – 1.83 % for wet season higher that the ash in the column chromatography and the modified products (Table 2) is less than that of Gum Arabic (2.94 – 6.56%. The difference in value is due to fact that the crude sample and the direct extracted samples may contain some impurities which may contribute to the increase in the amount of the ash.

*Moisture Content***:** The moisture content of crude exudate is 1.32 – 1.36%. It is within the same value in both the direct and the column extracts. The moisture content decreases from crude exudate to saponification product (1.00 - 1.10%) and to acid hydrolysis product (1.05 – 1.08%). The moisture content is low in all cases because of the resinous nature of the exudate but less than that of Gum Arabic (9.0± 0.58).

*Flash and Charring Temperature***:** The charring and flashing temperatures of crude exudate are between 190±7.9 to 200 ± 7.9 and 100± 7.9 110 ± 7.9 ^0C respectively in crude exudate and modified products. These temperatures, which indicate the thermal stability of crude exudate, are above the temperature encountered during processing, storage or normal handing of materials. The temperature reduces in both acid hydrolysis product and saponification product and less compared to the flash point of Gum Arabic (90± 2.3)

Table 3: Charring temperature (^oC)

Sample	Dry Season (^oC)			Wet Season (^oC)		
	ESD	IKSD	USD	ESD	IKSD	USD
Crude	200 ± 7.9	200 ± 3.4	200 ± 5.7	200 ± 7.9	200 ± 3.4	200 ± 5.7
Direct Extract	200 ± 7.9	200 ± 3.4	200 ± 5.7	200 ± 7.9	200 ± 3.4	200 ± 5.7
Chromatography column	200 ± 7.9	200 ± 3.4	200 ± 5.7	200 ± 7.9	200 ± 3.4	200 ± 5.7
Saponification Product	190 ± 5.7	190 ± 7.9	190 ± 3.4	190 ± 7.9	190 ± 3.4	190 ± 5.7
Acid Hydrolysis Product	190 ± 7.9	190 ± 3.4	190 ± 5.7	190 ± 5.7	190 ± 7.9	190 ± 3.4

Each data is a mean of three replicates determination ± SD

*Viscosity***:** The viscosity data is obtained for the exudate as reduced time of flow. This is similar to the reduced viscosities of grewia gum, trangacanth, acacia and methylcelluose (Okafor, 2001). The ability of exudate gum to increase the viscosity of solution is directly dependent on their molecular weight; hence the molecular weight of the exudate can be determined through viscosity measurement. This relationship was employed recently to obtain a molecular weight of 316,000 for grewia gum (Okafor, 2001). High intrinsic viscosity implies high molecular weight for polymers which are readily applied in suspersion (Chukwu and Nwankwo, 1991) and emulsion (Gaind *et al.* 1968) technology; similarly the binding properties of polymers used in tableting of drugs are dependent on molecular weight (Udeala and Chukwu, 1985). High molecular weight binding materials could prolong tablet disintegration, drug dissolution and bioavailability (Huber and Christenson, 1966). In this case, the rate of hydration and solubility of the drug is decreased. This concept is applied in sustained release formulation.

*Density***:** The densities of direct extract and column chromatography of crude exudate are 0.76$\pm$. 0.03 – 0.77$\pm$0.02g/cm^3 and 0.82 $\pm$ 0.01 – 0.89 $\pm$ 0.01 g/cm^3 respectively, while those obtained for acid hydrolysis product and saponification product are 0.94 $\pm$ 0.02 – 0.98 $\pm$ 0.01g/cm^3 and 0.88 $\pm$ 0.02 – 0.94 $\pm$ 0.04 g/cm^3 respectively. Density is a measure of packing characteristics of powered materials. An increase in density is good in tableting because of reduction in the fill volume of the die cavity of the tableting machine. The saponification product is much more crystalline due to added sodium ion (Na^+) with no impurities. In hydrolysis product, the density increased more than that of the crude exudate

Table 5: Densities of Exudate (g/cm^3)

n-hexane Extract	Dry Season (g/cm^3)			Wet Season (g/cm^3)		
	ESD	IKSD	USD	ESD	IKSD	USD
Direct Extract	0.76± 0.03	0.76± 0.01	0.76± 0.02	0.77 ±0.01	0.77± 0.02	0.77± 0.02
Chromatography column Extract	0.87± 0.01	0.84± 0.03	0.82± 0.01	0.89± 0.01	0.87± 0.01	0.88± 0.01
Saponification Product	0.90± 0.01	0.88± 0.02	0.88± 0.01	0.89± 0.01	0.92± 0.05	0.94± 0.04
Acid Hydrolysis Product	0.98± 0.01	0.97± 0.02	0.95± 0.03	0.94± 0.03	0.97± 0.02	0.98± 0.03

Each data is a mean of three replicates determination ± SD

Electrical Conductivity: Electrical Conductivity data (µS/cm) for the crude exudate, direct purified extract, column chromatography purified extract saponification product and acid hydrolysis product table 6, shows that the electrical conductivities in the direct purified exudate are similar while there is an increase in the values of the saponification and acid hydrolysis product. The conductivity could be explained in terms of the presence of some metal ions of K^+, Mg^{2+} and Na^+ as major charge carries in the exudate gum while the increase in both the saponification and acid hydrolysis product is due to the added ions from the reactants (H_2SO_4 and NaOH). Conductivity value is important to know because, if the exudate is used as a paper sizing agent during paper making, there is need to know how much such paper can conduct electricity in order to determine the application of the paper

Table 6: Electrical Conductivity data (µS/cm)

	Dry Season (µS/cm)			**Wet Season (µS/cm)**		
Sample (n-hexane extract)	ESD	IKSD	USD	ESD	IKSD	USD
Direct Extract	28.80	28.60	28.40	28.80	29.00	29.20
Chromatography column	28.60	28.50	28.40	28.70	28.60	28.50
Saponification Product	30.40	30.35	30.30	30.60	30.80	30.90
Acid Hydrolysis Product	32.40	32.20	32.10	32.80	32.90	33.00

*Conclusion***:** The physicochemical characteristic of *Dacryodes edulis* is found to be fluctuated with seasonal variations during the present investigation. The correlation coefficient showed positive relationship among the properties. The study reveals that exudate of *Daryodes edulis* can be tapped in large quantity in the dry season and is rich in lignin content but the seasons does not affect the properties. This study may be helpful in best utilization and sustainable management of the plant exudate for industrial uses in a paper industry.

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