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Article

Studies on the Industrial Application of Exudate from *Dacryoides edulis*

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Abstract: This study investigates the industrial applications of exudates from *Dacryoides edulis* (local pear tree). Solvent extractions of crude exudate gave yields of 24.3% in ethanol, 22.2% in petroleum ether and 27.5% in benzene, respectively. Separation of each extract by TLC indicated that each was a mixture of compounds, such as resin acids, lignin and carbohydrates. Treatment with hot dilute acid (10% H_2SO_4) and dilute alkali (10% NaOH) resulted in an aqueous layer, on top of which was an oily liquid that was solidified on cooling. Both the crude and solvent free extract burned readily in air, but the acid and alkali treated solid products did not. The saponified exudate was also found useful in sizing papers. Therefore, the plant exudates can be utilized as fuel and fire retardant as well as paper additive to control water penetration in paper.

Keywords: exudate; *Dacryoides edulis*; extraction; application; paper additive; fuel.

1. Introduction

The *Dacryoides edulis* (local pear tree) is a dioecious shade loving species of non-flooded forests in the humid tropical zone. It is a medium-sized evergreen tree reaching a height of 18-40 m in the forest (Verheij, 2002). *D. edulis* is a versatile plant in African ethnomedicine, as its various parts are employed to treat several diseases. The bark of the plant has long been used to treat wound, the stem and root are also used as chewing sticks for oral hygiene, while the leaves are employed to cure skin diseases, such as rashes, scabies, ringworm and external wounds (Ajibesin *et al*., 2008; Igoli *et al*.,

2005). The fruits and seeds of the plant are rich in oils (lipid), and some free fatty acids were reported to exhibit considerable nutritional value (Kinkela *et al*., 2006; Obasi and Okoli, 1993).

Exudates from the stem are viscous adhesive materials which ooze out from the injured stem of the plant. Resins or exudates occur in the genus and the resin from some species is used in African medicine (Leakey, 1999). The resin is medicinal and is applied to cure skin diseases such as ringworms, craw-craw and wounds. They are also used to treat parasitic organism like ticks and jiggars (Hutchinson *et al.,* 1963). The exudates are used in food and cosmetic industry as thickeners flavors, stabilizers and as emulsifying agents in drugs and cosmetics (Ekpa, 1993). Exudates from *D. edulis* when was applied in lotions and creams stabilize emulsion, add smooth to the skin and form protective coating on the skin. The exudates are used in traditional medicine as antibacterial agent and incense. It is believed that the smoke and sweet smell from the exudates when burning wades off evil spirit (Sofowara, 1993).

The traditional history has it that the exudates has been in used as fuel from the ancient times and must have been discovered when the stem of the dead plant was used as firewood. The exudates which solidifies into a dark mass on drying is still used as fuel in locally designed lamps especially in the villages. Naturally as an adhesive, it is also used as gum.

Since the paper makers' rosin is an extract from the stem of *Pinus caribeae* (Casey, 1980), it also becomes necessary to study the possibility of application of these pear plant exudates as a paper sizing agent. Therefore in this work some properties of this exudates and its application as paper sizing agent and as a raw material for candle making have been studied.

2. Materials and Methods

2.1. Materials

The crude exudates were obtained by making knife cuts on about 1 mm deep into the sap wood. The viscous fluid oozed out and was collected into a glass container, and hardened samples from previous cuts were scrapped off from the stem too. For comparison as paper sizing agent exudates from the plant *pinus caribeae* from where the commercial sizing agent rosin is obtained was also prepared. Commercial rosin size was obtained from the Nigerian Newsprint Manufacturing Company, Oku Iboku in Akwa Ibom State, Nigeria for future comparison.

2.2. Purification of the Crude Exudates

Crude exudates were found soluble in hot ethanol, petroleum ether, benzene and n-hexane and very sparingly soluble in hot water. Soxhlet extraction was then carried out, the solvent evaporated and the yield determined.

2.3. Properties of the Exudates

Rosin (Liebermann-Starch) test: lignin test was done using phloroglucinol and iodine test for the starch were carried out according to Browing (1977).

2.4. Acid and Alkaline Treatment

The 2.0 g of each sample was separately boiled in 10% H_2SO_4 and 10% NaOH in a beaker. The commercial saponified rosin was treated with 2 M H_2SO_4 to free the resin acid.

2.5. Separation by Thin Layer Chromatography

The extracts and purified commercial rosin were spotted on the chromatography plate prepared from silica-gel and eluded with 2:2:1 petroleum ether, benzene and ethanol mixture and developed in iodine vapor, and the resolution factors (RF) are obtained.

2.6. Industrial Application of the Exudates

2.6.1. Preparation of Exudate Size Stock

The 3% solution of saponified exudates from *Dacryoides edulis* and *Pinus caribeae,* the commercial rosin were separately prepared in water, and 3% aluminum sulfate dihydrate [$Al_2(SO_4)_3.2H_2O$] solution and 3% starch emulsion were also made.

2.6.2. Application as Paper Sizing Agent

Handmade sheets were formed from 0.3% consistency stock made from unbeaten bleached virgin kraft pulp using locally designed hand mould. The following sheets were made:

(i) Water leaf sheets with no additives

(ii) Starch sized sheets containing only starch

(iii) Commercial rosin (sizing agent) sized sheets

(iv) Exudate sized sheets containing 3 cm^3 of 2% saponified ethanol exudates extract with 3 cm^3 of 3% $Al_2(SO_4)_3.2H_2O$ and 5 drops of 3% starch emulsion, both the saponified *Dacryoides edulis* and *Pinus caribeae* ethanol extracts were used.

Determination of hand sheet dimensions: The weight, the length and the width (caliper) were determined with the mettle balance, meter rule and the micrometer screw guage, and the area and grammage (weight in grams per square meter) were calculated.

Water absorbent capacity: Each dry sample sheet was cut into 3 cm x 3 cm pieces. Two pieces were weighed separately and later stapled together and reweighed. The dry stapled pieces of paper

were inserted in distilled water contained in a beaker for 15 seconds to absorb water, and it was then removed and the excess water cleaned off on the side of the beaker for another 15 seconds. The weight of the stapled wet pieces was immediately taken again and the difference between the dry weight and the wet weight is the amount of water absorbed.

$$Absorbent\ capacity = \frac{amount\ of\ water\ absorbed\ by\ sheet}{weight\ of\ the\ \ dry\ sheet}$$

2.6.3. Preparation of Candle from Exudate Wax

Hexane exudates extract was melted in a beaker and heated to remove the solvent. The melted waxy material was poured into a laboratory mould designed for this purpose. The system was kept for 24 h at room temperature for the candle to be hardened. The cylindrical candle obtained was subjected to flame test in comparison with commercial petroleum wax candle. The time for burning 3 g of each sample was recorded as the candle power.

3. Results and Discussion

The characteristics of the exudates studied were those which were related to their intended application as paper sizing agent and fuel. Elements composition and electrical conductivity of the exudates of *Dacryoides edulis* (local pear) were studied by Ekpe *et al.* (1993), while detail properties of exudates from *Pinus caribeae* (pine, Christmas tree) have been reported in Britt (1981) and Casey (1980).

The exudates were soluble in ethanol, petroleum ether, benzene and hexane. Purification of the light brown crude pear exudates yielded 22.2% brown extract in ethanol, 24.3% light brown extract in petroleum ether and 27.5% brownish yellow extract in benzene, respectively (Table 1). On the other hand, these solvents ethanol, petroleum ether and benzene extracted 5.5%, 1.9% and 2.74% exudates from the wood of *Pinus caribeae*, respectively. The hot acid treatment yielded only oily material which solidified on cooling. This solid is a mixture of unsaponifiable waxy matter mixed with resin and some fatty acids.

Table 1. Yields of the Solvent Extract

Solvent	**Yield (%)**	
	Dacryoides edulies	***Pinus caribeae***
Ethanol	22.2	5.5
Petroleum ether	24.3	1.9
Benzene	27.5	2.7

On ignition the crude exudates burned with sooty flame and a peculiar smell. The hexane extracted exudates solidified on evaporation of the solvent and also burned with less sooty flame when ignited. It was this hexane extracted pear exudates that was used in making cylindrical candle 12 cm in length and 1.5 cm in diameter. The candle was grey in color and burned at the rate of 2 min per gram as against 1min per gram by the commercial petroleum wax candle. The pear exudates candle gave good illumination but produced more soot than the petroleum wax candle. It is hoped that more purification and chemical modification will improve both the quality of the flame and reduce its rate of burning.

Table 2 shows the result of elution of the extracts on silica gel thin layer chromatographic plate with volume ration 2:2:1 of petroleum ether, benzene and ethanol mixture, the Retention Factors (RF) are shown.

Table 2. RF of exudate extracts on silica gel with volume ratio 2:2:1 of petroleum ether, benzene and ethanol eluent system

Exudate Extract	**RF**		
	***Pinus caribeae* exudate**	***Dacryoides edulies* exudate**	**Rosin (Control)**
Ethanol	0.31	0.31	0.35
	0.51	0.51	0.55
	0.81	-	-
	1.00	-	-
Petroleum	0.47	0.39	0.48
	0.73	0.58	0.87
Benzene	0.48	0.58	0.26
	0.76	-	0.43
	-	-	0.67

The ethanol extracted exudates from the pear tree and the pine compared well with those of the commercial rosin (sizing agent), indicating that their components are similar, being mainly resin acids consisting mostly of abietic and neoabietic acids. Therefore ethanol extracted exudates was used as paper sizing agent.

Table 3 shows the grammage and the water absorbent capacity of the hand sheets formed in the laboratory and sized with aqueous solution (3%) of the saponified exudate, and water leaf sheet and commercial resin sized sheet served as control. The result shows that the water leaf sheet had the highest water absorbent capacity while the sheets treated with pear exudates extract were better sized than both the commercial rosin and pine extract treated sheets. Therefore the exudates of pear tree can be used as paper sizing agent for controlling moisture penetration into sheets of papers, especially

when writing or drawing with aqueous ink. Paper sizing entails the blocking of the several openings present in the paper sheet and the covering of the highly hydrophilic cellulosic fibers that make up the paper structure with substances which can induce some extent of hydrophobicity and prevent capillary rise in paper sheets. The sizing agent may be retained by either intermolecular forces of attraction or covalent bonds depending on the type of the sizing agents. In the case of rosin size, $Al_2(SO_4)_3$ serves as the retention aid, and the structure of the size on the paper is as suggested by Akpabio (1985).

Table 3. Water absorbent capacity of sized sheets

Sheet Sample	Grammage (gsm)	Absorbent Capacity (g/g)
Water leaf	73.22	0.63
Starch sized	73.25	0.54
Commercial rosin sized	73.00	0.53
Pine exudates sized	73.15	0.47
Pear exudates sized	73.21	0.46

4. Conclusion

The exudates from *Dacryoides edulis* is suitable for lighting purposes as a source of fuel especially in case of scarcity of petroleum fuel (kerosene) and has been found to be a superior sizing agent to control water penetration in paper. Unlike the extraction of commercial rosin size from the pine tree *(Pinus caribeae)* after it has been cut down, the pear exudates can be tapped directly from the living tree which continues to produce the edible fruits at the same time. Therefore exploitation of this economic tree, *Dacryoides edulis* for the production of exudates which can be used for the production of the candle wax for lighting purposes and modified into paper sizing agent, is of great economic importance, more investigations are being carried out to improve these products for commercial purposes.

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