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Development of an Adhesive from *Prosopis africana* Seed Endosperm (Okpeyi)

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***Abstract*—**This research work is an experimental study, through development of an adhesive from *Prosopis africana* endosperm. The prosopis seed for this work were obtained from Enugu State in the South East part of Nigeria. The seeds were prepared by separating the endosperm from the seed coat and cotyledon. Three methods were used to separate them, which are acidic method, roasting method and boiling method. 20g of seed were treated with different concentrations (25, 40, 55, 70, and 85% w/w) at 100°C and constant time (30 minutes), under continuous stirring with magnetic stirrer. Also 20g of seed were treated with sulphuric acid of concentrations 40% w/w at 100°C with different time (10, 15, 20, 25, 30 minutes), under continuous stirring with magnetic stirrer. Finally, 20g of seed were treated with sulphuric acid of concentrations 40% w/w at different temperature (20°C, 40°C, 60°C, 80°C, and 100°C) with constant time (30 minutes), under continuous stirring with magnetic stirrer. The whole endosperm extracted was adhesive. The physical properties of the adhesive were determined (appearance, odour, taste, solubility, pH, size, and binding strength). The percentage of the adhesive yield makes the commercialization of the seed in Nigeria possible and profitable. The very high viscosity attained at low concentrations makes prosopis adh*esive* an excellent thickener in the food industry.

***Keywords*—**Endosperm, adhesive, ethanol, *Prosopis africana* seed.

I. Introduction

PROSOPIS trees or shrubs are woody perennials belonging to the family *Leguminosae*. The Prosopis genus comprises about 44 species of trees and shrubs; the number could be as high as 77 since similar species are now included in other genera like *Acacia* [1]. The species have been divided into five sections, distributed in North America, Central/South America, Africa, and Asia. Africans called it *Prosopis africana*. It occurs naturally in arid and semi-arid areas where it has been used by local populations as a good source of timber, fuel, fodder, food, gum, tannin or dyestuff and fish poison. In Nigeria, it is predominant in North Central geo-political zone, around Kogi, Benue, Nasarawa, Kaduna, Niger and Platue states. It is also sparsely growing in the South-East geo-political zones.

The pod of mesquite (*Prosopis* spp.) consists of three separable components: exo and mesocarp (pulp), endocarp and the seed [2], [3]. The pods are used as food for cattle and fish poison by the fishermen. Interestingly, these pods also contain seeds which can be processed to produce local food seasoning "okpeyi" [4], [5], while the seed coat with the endosperm is thrown away as waste. However, it has been observed that the water used in the processing of the boiled seed becomes more viscous and exhibit adhesive or gummy character. The viscosity of the processing water was attributed to the dissolution of the endosperm of boiled seed. This has been supported by work carried out by [6], [7]. It has also been reported that adhesive can be produced from other species of *Prosopis* [8] in which adhesive is extracted from seed endosperm. Several leguminous seed contain adhesive polysaccharides in the endosperm, which yield highly viscous aqueous solutions and therefore find industrial applications as thickeners. The three major adhesive currently used by the food industries are *Prosopis* adhesive (guar gum), locus (carob gum) and tar adhesive.

A complete separation of seed coat from the endosperm of *Prosopis africana* has been carried out by thermal, physical and chemical processes. However, this research work will adopt the chemical process by using sulphuric acid. In doing this, various concentrations of the acid are used at a fixed temperature while monitoring the yield of endosperm at those concentrations of acid and also at various times with the aim of determining the optimum conditions for producing endosperm for adhesive formulation. Formulation of adhesive from the waste is very beneficial as it will guarantee Nigerians safer environment and employment creation.

II. Materials and Methods

In order to achieve the objective of this research work, "developing of an adhesive from *Prosopis africana* endosperm" certain laboratory work was carried out. This research work adopted the following steps in development adhesive from *Prosopis africana* endorsperm; source of raw material, equipment, processing of raw material (technological processes for endosperm separation), preparation of gum solution, and test of the gum formed.

Sourcing of Raw Material: Prosopis africana seeds were bought from the market in Enugu State in the South East part of Nigeria. Bought seeds were washed to eliminate adhering sand. Immature seeds were carefully sorted out by handpicking. There was no broken seed due to the hardness of the seed coat.

Equipment/Apparatus: flat bottom flask, stop watch, measuring cylinder, electrical weighing balance, magnetic stirrer, beaker, spatula, tensometer, mechanical shaker, heating mantle, viscometer, magnetic bar or rod, thermometer, and rotating furnace.

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Reagent: Tetraoxosulphate (vi) acid (H_2SO_4), distilled water, formaldehyde (methanol), and sodium hydroxide.

Processes for Endosperm Separation: are acidic process, roasting process and boiling process.

Acidic Process Separation: In a set of experiment, 20g of seed were treated with difference concentrations (25, 40, 55, 70 and 85% w/w) at 25^0C and constant time (30 minutes), under continuous stirring with magnetic stirrer. After stirring, sulphuric acid was quickly and completely drained and then the seeds were extensively washed with tap water. Special attention was paid to this step to prevent excessive local heating by acid-water interactions, which could cause seed damage. During the washing operation, the seeds were manually rubbed to eliminate the burned coat fragments.

The "peeled" seeds were dried in an oven at 60^0C and then cracked to get endosperm splits. Separation of the splits from the cotyledon fragments was tried by sieving (10 mesh ASTM) and air streaming. However, a final manual selection was necessary to obtain pure splits. The pure endosperms were crushed to obtain dry adhesive. The crushed adhesives were then dissolved in de-ionized water. The pH and the viscosities were determined. Graphs of concentration against yield (Fig. 1), concentration against binding strength (Fig. 4), and concentration against pH (Fig. 5) were all plotted Also, 20g of seed were treated with sulphuric acid of concentrations 40% w/w at 100°C with different time (10, 15, 20, 25, 30 minutes), under continuous stirring with magnetic stirrer. The step above later followed and a graph of yield against time (Fig. 3) was plotted.

Roasting Process: A total of 100g of seeds were used for the roasting trials. The seed to be roasted were not absolutely clean but contained approximately 5% of exo- and endocarp fragments.

The splitting procedure starts with roasting of the seeds. All seeds are heated for several minutes. During the roasting process, the endosperm remains intact and flexible, while the seed coat and the cotyledon, which are more sensitive to heat, become brittle. The seed coat and the cotyledon are separated from the endosperm split by sieving. Remaining traces of seed coat and the cotyledon on the endosperm split. Particles are finally removed through a series of physical cleaning steps. A composite sample is examined to ensure the material meets the specifications for splits intended for use in the production of refined adhesive.

Boiling Process: A 200g of seed were used. The seed were boiled with water in a heating mantle of 90°C for 11 hours. The boiled seed were cracked to separate the endosperm from the seed coat and the cotyledon by manual pressing. The obtained endosperms were dried in an over of 90°C. The dried endosperms were crushed to obtain dry adhesive.

A. Preparation of Adhesive Solution

The solution of the adhesive were prepared by weighing the yield of the dry adhesive in a beaker and 250ml of de-ionized water were slowly added under continuous stirrer in a magnetic stirrer for 1 hour to avoid formation of lumps under 50°C. The solutions were then decanted to obtain adhesive solution. The gum solutions were stored at 4°C in a covered beaker. De-ionized water was added to the gum solution to correct the evaporation losses.

B. Measurement of the Viscosity of the Adhesives Formed

The viscosity of the adhesive formed of different concentrations such as (25, 40, 55, 70, and 85% w/w) were measured at 25°C using Brookfield viscometer.

C. Measurement of pH of the Adhesive

The pHs of the adhesives were measure using pH meter ranges from 0-15. 2m of NaOH were added to different concentration of the adhesive under continuous stirring, to obtain an alkaline medium.

D. Determination of Binding Strength of the Adhesive

The binding strength of the adhesive was carried out with five cartons of 10cm, 2cm width. The five cartons were papered by applying the *Prosopis* adhesive over an area of the five cartons and left for 24 hours. The joined cartons were taken to mechanical engineering section where tensometer machine was used to determine the binding strength of each adhesive.

III. Results and Discussion

This project work was successful. Based on the analysis done and other on the process involved in the production of adhesive from endosperm of *Prosopis* seeds, the result gotten are shown in Tables I–VI and Figs. 1–5.

TABLE I
PHYSICAL PROPERTIES OF THE *PROSOPIS* ADHESIVE PRODUCED

Appearance	Off-white fine powder
Odour	Neutral
Taste	Neutral
Particle size <250 micron	≥ 99%
Solubility	Insoluble in ethanol; disperses well in cold water and forms a colloidal solution

TABLE II
THE EFFECT OF CONCENTRATION ON THE YIELD OF *PROSOPIS* ENDOSPERM AND THE VISCOSITY OF THE *PROSOPIS* ADHESIVE FORMED

Concentration (%w/w)	Yield (g)	Viscosity (pa/s)
25	1.82	0.13
40	2.46	0.18
55	0.82	0.14
70	0.37	0.10
85	0.2	0.02

TABLE III
THE EFFECT OF TIME ON YIELD OF THE *PROSOPIS* ENDOSPERM AND THE *PROSOPIS* ADHESIVE FORMED

Temperature (^{0}C)	Yield (g)	Viscosity (pa/s)
20	4.5	0.28
40	3.5	0.24
60	2.9	0.20
80	2.49	0.18
100	2.45	0.18

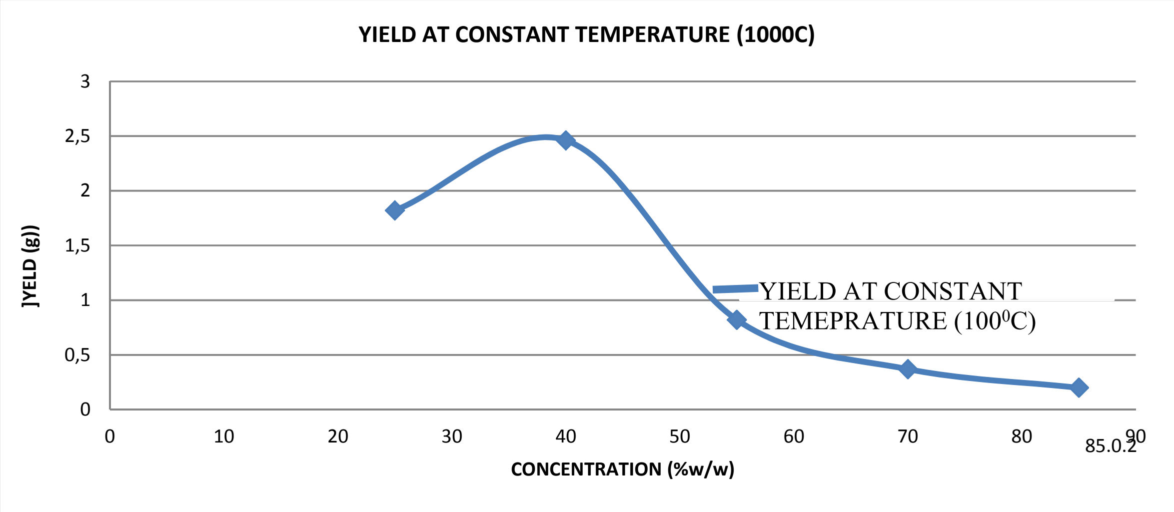

Fig. 1 The Effect of Concentration on the Yield of *Prosopis* Adhesive

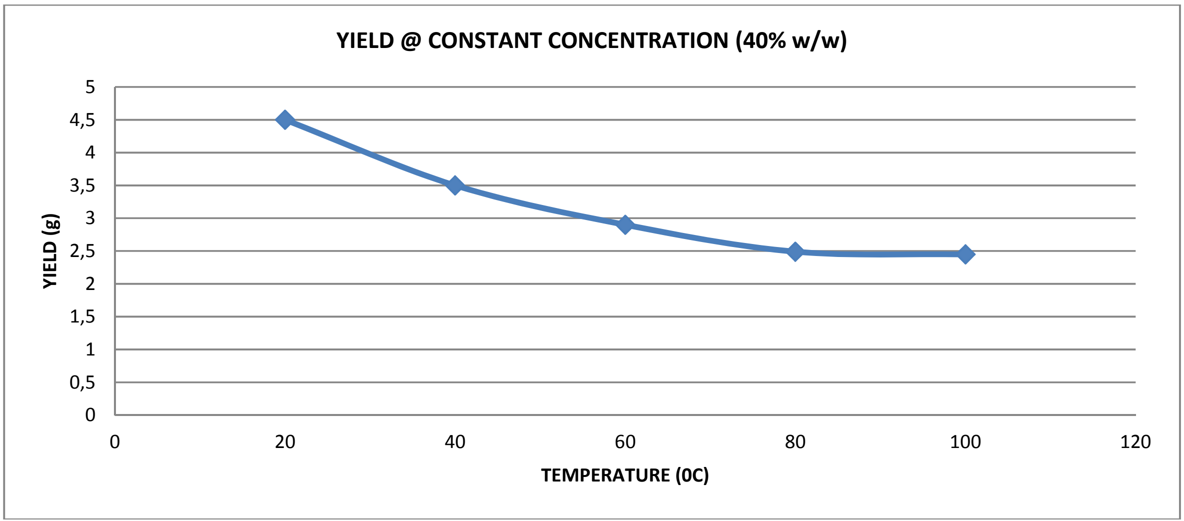

Fig. 2 The Effect of Temperature on the Yield of *Prosopis* Endosperm

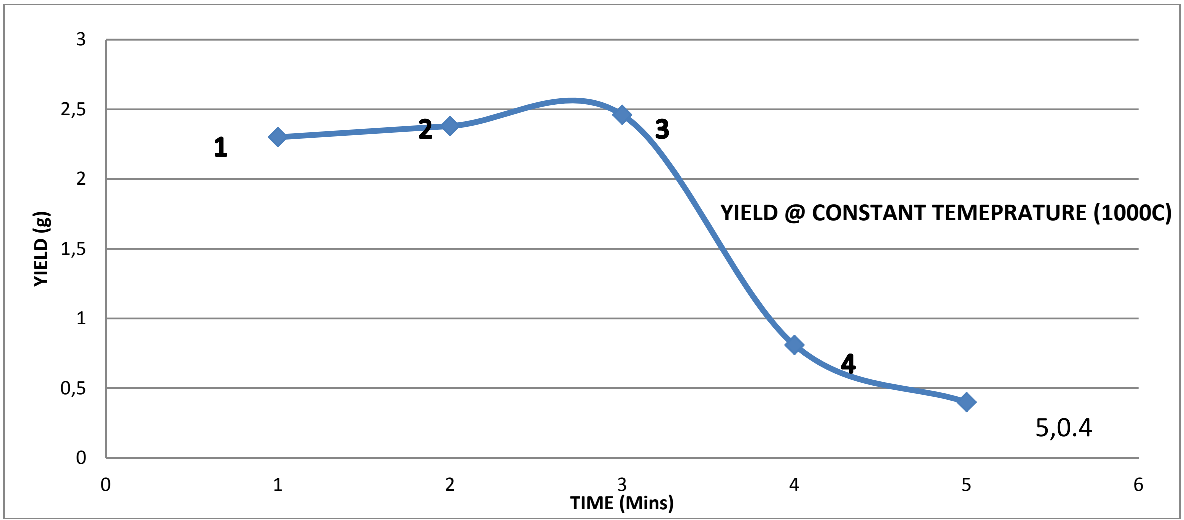

Fig 3 The Effect of Time on Yield of the *Prosopis* Endosperm

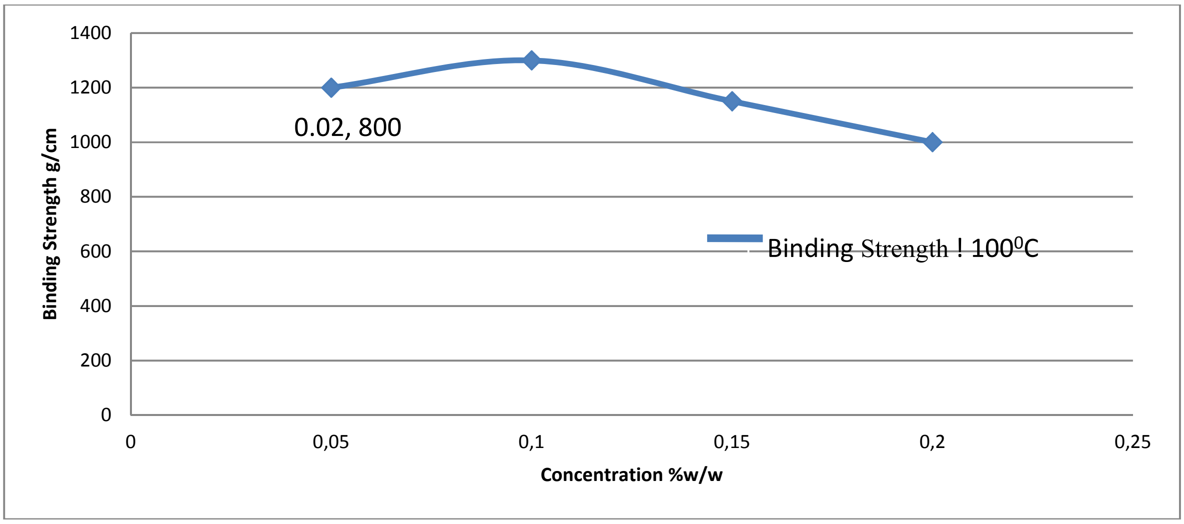

Fig. 4 Effect of Concentration on Binding Strength of the Adhesive

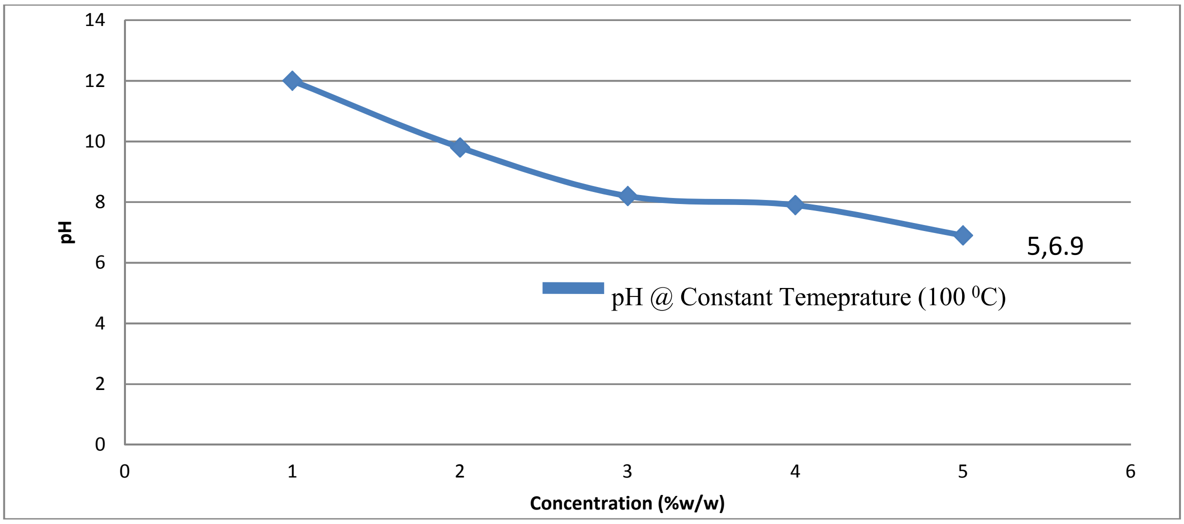

Fig. 5 Effect of Concentration on pH of the Adhesive

Prosopis adhesive attained at low concentration in water provides high viscous solution. The viscosity of the 25% w/w *Prosopis* adhesive was higher than that of 40% w/w. In addition, 40% w/w of the *Prosopis* adhesive was higher than the 55% w/w likewise 70%w/w and 85%w/w. Therefore, decrease in concentration increases the yield of the *Prosopis* endosperm formed and viscosity of the *Prosopis* adhesive.

Temperature causes the *Prosopis* adhesives to lose their viscosity properties. The result of this study shows that the viscosity of 40%w/w was decrease when the *Prosopis* seed were heated and held in the physiological temperature for 30 minutes e.g. for 20°C, 40°C, 60°C, 80°C and 100°C. Therefore, increase in temperature decreases the viscosity of the *Prosopis* adhesive.

Time causes the *Prosopis* endosperm to reduce in size and the *Prosopis* adhesive that is formed to lose their viscous properties. The result of this study that the viscosity of *Prosopis* seed was decrease when 20g of the seed were treated with sulphuric acid of concentrations 40%w/w at 100^0C with different time (10, 15, 20, 25, 30 minutes), therefore increase in time decreases the viscosity of the gum and the yield of the *Prosopis* endosperm.

TABLE IV
THE EFFECT OF TIME ON YIELD OF THE *PROSOPIS* ENDOSPERM AND THE *PROSOPIS* ADHESIVE FORMED

Time	Yield (g)	Viscosity (pa/s)
10	2.3	0.18
20	2.38	0.19
30	2.46	0.15
40	0.81	0.14
50	0.4	0.12

From the result of the study, the binding strength of the *Prosopis* adhesive increases as the viscosity increases, therefore, increase in viscosity increases the binding strength of the *Prosopis* adhesive. In addition, the breaking point of the adhesive is 1300g.

TABLE V
THE EFFECT OF CONCENTRATION ON BINDING STRENGTH OF THE ADHESIVE

Concentration (%w/w)	Viscosity (pa/s)	Binding Strength of the Adhesive g/cm^2
25	0.13	1200
40	0.18	1300
55	0.14	1150
70	0.10	1000
85	0.02	800

TABLE VI
EFFECT OF CONCENTRATION ON PH OF THE ADHESIVE

Concentration (%w/w)	Viscosity (pa/s)	pH
25	0.18	12
40	0.18	9.8
55	0.14	8.2
70	0.10	7.9
85	0	6.9

From the result and Fig. 5, the pH of the *Prosopis* adhesive produce is alkali. The alkalinity of the *Prosopis* adhesive decreases as the concentration of the acidity of the adhesive increases. Therefore, the increase in the concentration of the acidity of the adhesive increases, the alkalinity of the gum decreases. In addition, it was observed that:

1. Sulphuric acid darkens the seed coat
2. Ethanol removes sulphuric acid (H_2SO_4) in the *Prosopis* seed but also reduces the gumming properties of the adhesive.
3. Sieving the crushed adhesive to powder helps to obtain a fine texture adhesive without coarse particle, since adhesive with coarse particles produce very poor joints between two adherent and substrates.

The use of formaldehyde (methanol) as preservatives protects the adhesive from bacterial activities and increases water holding capacity, curing time and useful glue life.

It is important to note that small quantity of methanol should be added otherwise the viscous properties will be lost. In addition, it was observed that the gum gotten from acidic process has more viscous property than the ones gotten from roasting and boiling method because the weight of a single endosperm from acidic process is higher than that of the roasting and boiling method.

IV. CONCLUSION

Having carried out investigations on the development of adhesive (gum) and some properties of the *Prosopis* adhesive, the results of the test has shown that the acid decoating process has proven to be effective but strongly dependent on the stirring conditions. The best results were obtained by treating 20g of seeds with 40ml of (%w/w) H_2SO_4, at 20°C for 30 minutes and stirring at 100rpm. The physical properties of the gum formed are good data source useful in many industries.

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