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Impacts of viscosity enhancer and gelatinization modifier on physicochemical properties of starch-based adhesive obtained from cassava *(Manihot Esculenta)*

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ABSTRACT

Aim: This study was aimed to determine the impact of borax and gelatinization modifiers (NaOH) in the physicochemical properties of starch-based adhesives obtained from cassava.

Method and Materials: Different weights of Sodium tetraborate (borax) and concentrations of Sodium hydroxide were used as viscosity enhancer and gelatinization modifier respectively in the preparation of starch-based adhesive from cassava (*Manihot esculenta*) to ascertain their impact on the physicochemical properties of the adhesive produced.

Results: The viscosity, density, bond strength of the adhesive obtained from cassava (*Manihot esculenta*) increased with increase in the weight of borax and concentration of sodium hydroxide. Drying time of starch-based adhesive obtained from cassava (*Manihot esculenta*) was inversely proportional to the weight of borax and concentration of NaOH respectively. The addition of a gelatinization modifier (NaOH) with molar concentration of 0.3 M and a viscosity enhancer (borax) with a weight of 15 g to a starch adhesive from cassava (*Manihot esculenta*) can produce an adhesive with acceptable bond strength.

Conclusion: It was concluded that higher viscosity of starch-based adhesive obtained from cassava (*Maniholt esculenta*) the better rheological formulations of the adhesive and it is directly proportional to the ability of the adhesive to maintain stability of its molecular composition during curing (gel formation) which subsequently determines the bond strength of the adhesive.

Keywords: Adhesive, amylose, concentration, rheology, vegetable glue, weight.

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Introduction

Diabetes Vegetable glue may be defined as a homogeneous mixture of starch, water and solvent from amyl cellulose such as caustic soda, with the appearance and adhesive properties of animal glue. Starch obtained from cassava (*Manihot esculenta*) generally forms the basis of this material, the water content varies from 2 to 4 times the weight of the starch and the caustic is generally about 5 per cent and may vary between 2 and 12 per cent of the weight of the starch. Cassava (*Manihot esculenta*) is a tropical crop (Fig 1) that supplies between 200 and 1000 calories to some 700 million people worldwide daily (Udoudoh, 2011; Irtwange and Achimba, 2009).

Nigeria is ranked first among major producers of cassava in the world, producing about 33 million metric tonnes of cassava in 1993 alone. Over 95% of cassava produced in Nigeria is used as food for the country's teeming population, being one of the country's staple foods (Onwuka and Ogbogu, 2007). Starch is one of the major derivatives of fresh cassava roots, it is also a by-product of *garri* processing, and it accounts for 30 – 35% of fresh cassava roots. *Garri* is a dry, crispy, creamy-white and granular product obtained from cassava roots that have been crushed into a mash, fermented, sieved into small pieces and fried or roasted. The isolation of starch from fresh cassava roots is normally carried out in aqueous medium (Aviara et al., 2010).

One of the industrial applications of cassava starch is in the manufacture of natural adhesives. Natural adhesives are substances capable of binding materials together by attaching to the surface with the capacity of sustaining the designed load requirement (Oghenejoboh, 2012).

Natural adhesive includes animal glues, casein glues and vegetable glues made from starches and dextrin. There are several advantages obtained from using starch as a raw material in the production of adhesives, these advantages include its renewability, biodegradability, abundance, cost, stability in price and general friendliness to the environment. Starch can give adhesives an excellent affinity to polar substrates owing to its polymeric and poly-hydroxy properties (Gunorubon, 2012).

Among all the raw materials used for the manufacture of natural adhesives, cassava starch has distinct advantages over others (i.e. animal glues, vegetables and other root crops) because it is obtained from by-products that are normally discarded during traditional processing of *garri*, hence making its production cost effective. Cassava has a fine, smooth texture, non-staining and non-poisonous nature which makes it the best choice for domestic and most non-structural utilization. In addition, cassava-based adhesives are tackier, more viscous and their joints exhibit higher tensile strength than those from cereal and other tuber crops (Ojewumi et al., 2021). Another advantage of cassava-based adhesive is the temperature at which it turns into gel which ranges between 49 ^oC and 70 ^oC when compared to 62 ^oC - 73 ^oC for cereals such as corn, hence less heating is required to gelatinize cassava starch leading to greater preservation of energy (Masumbu et al., 2003). Gelatinization modifiers play important roles in determining the gelatinization temperature. The use of Borax ($Na_2B_4O_7.10H_2O$) is very essential in improving the adhesive characteristics of adhesives. Borax ($Na_2B_4O_7.10H_2O$) increases the viscosity, cohesiveness, tack characteristics of natural adhesives thereby enhancing their thermal, mechanical and adhesive performance (Masamba et al., 2001). The environmental friendliness of starch-based adhesives is being altered by the quantity of borax and gelatinization modifiers used during its manufacture as these chemicals have been proven to cause series of health problems in humans, animals and vegetation. Borax when ingested can cause vomiting, diarrhea, irritation to skin, eyes, nose throat and lungs when inhaled (Sanares et al., 2001). Other health hazards caused by borax incude hormonal issues, organ damage and even death in infants after prolonged exposure (Malinee, 2009). The aim of this study was to determine the impact of borax and gelatinization modifiers (NaOH) in the physicochemical properties of starch-based adhesives obtained from cassava. The physicochemical properties of adhesives play very important roles in the adhesive strength of adhesives. This study therefore unveils the minimum quantity of viscosity enhancers and gelatinization modifiers essential in obtaining the most desirable physicochemical properties in starch-based adhesives while still maintaining safety of humans and environment as well as saving cost.

Materials and Methods

Sample Collection / Preparation

Cassava tubers obtained from a community in Obio Akpor local government area of Rivers State, Nigeria was peeled, washed with water and then grated into a paste. The grated paste was soaked in water for two days to allow for partial fermentation, the mixture was then sieved using a mesh size of 50 µm. The filtrate obtained was allowed to settle in order to obtain coagulated starch slurry. The starch particles obtained was then spread on a clean stainless-steel plate and placed in an oven for 25 minutes at 100 ^0C to obtain dry cassava starch. To prepare the starch-based adhesive, 10 g of dry starch obtained was dissolved in different concentrations of 100 ml NaOH solution acting as a gelatinization modifier solution and then stirred continuously using a magnetic stirrer at 250 rpm. Sample was allowed to heat at the required reaction temperature and then the required concentration of borax ($Na_2B_4O_7.10H_2O$) which acts as a viscosity enhancer was added while stirring until a sticky product is obtained. The starch adhesive produced was allowed to dry at different drying times depending on the concentration of the viscosity enhancer and then cooled for the determination of different physicochemical parameters (Olomo 2022; Ozemoya et al., 2007).

pH Determination of Starch Based Adhesive:

The pH meter and associated electrodes were standardized using two reference buffer solutions within range of anticipated sample pH. The sample measurement was made under strict controlled conditions and prescribed techniques before the solidification of the sample after introduction of viscosity enhancer. The already calibrated electrodes were immersed into the sample.

Fig 1: Cassava (*Manihot Esculenta)*, Fig. 2: Milliliter Depth gauge and Fig 3: eXpert 5000 modular testing machine

As soon as the electrode output stabilizes, the stability indicator appears displaying the pH and temperature (ASTM D1583, 2018).

Determination of Density and Kinematic Viscosity:

The cells of the equipment were thoroughly cleaned. 2 ml of the test sample was introduced into the equipment through a connector installed for filling samples into the measuring cell which can be achieved either automatically or manually with the use of a suitable syringe after proper agitation of the test sample. On stabilization, density, kinematic viscosity and dynamic viscosity readings were displayed and the readings recorded (ASTM D4052, 2022)

Determination of Film Thickness of Starch Based Adhesive:

The film thickness of the starch-based adhesive was determined using a milliliter (mil) depth gauge (Fig 2). A mil depth gauge is a flat, metallic comb that easily allows operators to measure the thickness of a liquid coating before applying to a surface. The mill depth gauge was placed directly against the glue roller at 90^0 until the gauge makes contact. Wait for 5 – 7 minutes to let the notches become coated with the sample being measured, withdraw the gauge vertically. Read the gauge by examining which notches have become coated by the sample. The wet film thickness (WFT) of the sample is between the values of the smallest uncoated notch and the largest coated notch (Oghenejoboh, 2012).

Determination of Drying Time of Starch-Based Adhesive:

The drying time of an adhesive is determined by calculating the time immediately after applying the adhesive on a surface to the time a complete dryness is observed by intermittently touching the applied surface gently after every 10 seconds. Dryness is confirmed when the gummy or sticky nature of the adhesive vanishes *(*Aviara et al., 2010).

Determination of the Bond Strength of Starch-Based Adhesive:

The bond strength of the starch-based adhesive was determined with an eXpert 5000 modular testing machine (Fig 3). The prepared adhesive was bonded to the substrate material at a depth of approximately six inches with the free end of the sealant folded to a 180^0 angle. The free end of the substrate material in this case is wooden was attached to the vice grip on the crosshead of the testing machine with the free end of the adhesive secured to the second vice grip. The crosshead of the machine was moved at a specified rate after the adhesive sample has been inserted into the plane of the vice grips and the machine zeroed. Record the average load which is used to calculate the bond strength (*ASTM D903 2017).*

Results and Discussion

Results were recorded as impact of gelatinization modifiers and viscosity enhancers on the physicochemical properties of starch-based adhesive (Table 1) and impact of gelatinization modifiers and viscosity enhancers on the adhesive characteristics of starch-based adhesive (Table 2). Plot of viscosity of starch-based adhesive to weight of borax (Fig 4), pH (Fig. 5), Density (Fig. 6), Wet film thickness (Fig. 7) and bar chart illustrating the impact of gelatinization modifiers and viscosity enhancers on the adhesive characteristics of starch based adhesives (Fig. 8).

Table 1: Impact of gelatinization modifiers and viscosity enhancers on the physicochemical properties of starch-based adhesive

Conc. of NaOH (M)	Weight of Borax (g)	Viscosity (Pa.s)	pH	Density(g/cm^3)	Wet film thickness (mm)
0.100	5.000	82.52	8.90	1.017	0.200
0.200	10.000	89.81	8.70	1.022	0.200
0.300	15.000	98.55	9.47	1.024	0.300
0.400	20.000	103.28	9.20	1.025	0.500
0.500	25.000	110.03	9.50	1.032	0.500

Table 2: Impact of gelatinization modifiers and viscosity enhancers on the adhesive characteristics of starch-based adhesive

Conc. NaOH (M)	Weight of Borax (g)	Bond Strength (MPa)	Gelatinization Temp (^0C)	Drying Time (mins)
0.100	5.000	3.800	65.000	5.300
0.200	10.000	4.300	75.000	5.250
0.300	15.000	7.500	60.000	5.200
0.400	20.000	8.800	55.000	4.800
0.500	25.000	10.700	50.000	4.200

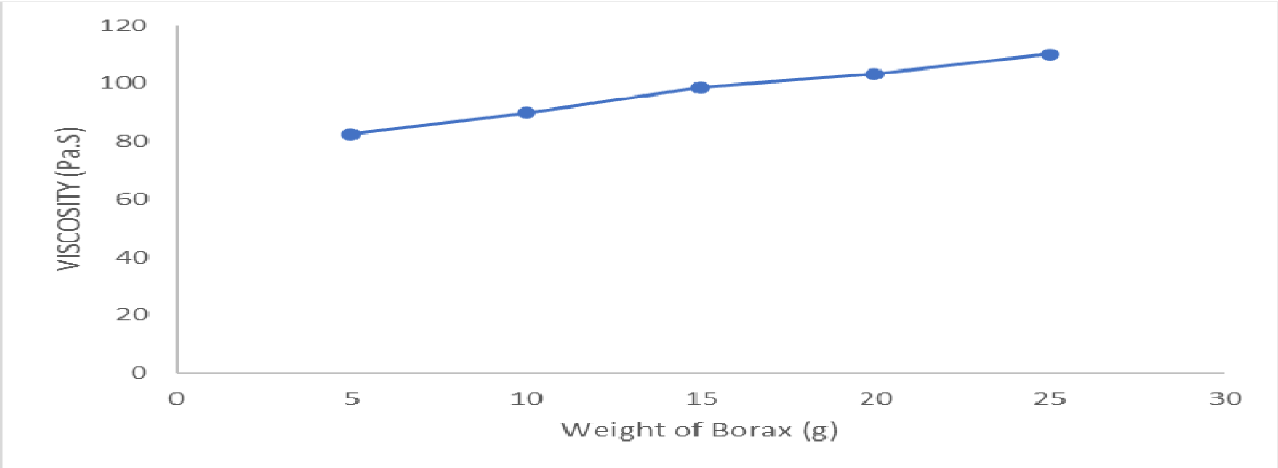

Fig 4: Plot of Viscosity of starch-based adhesive to weight of borax

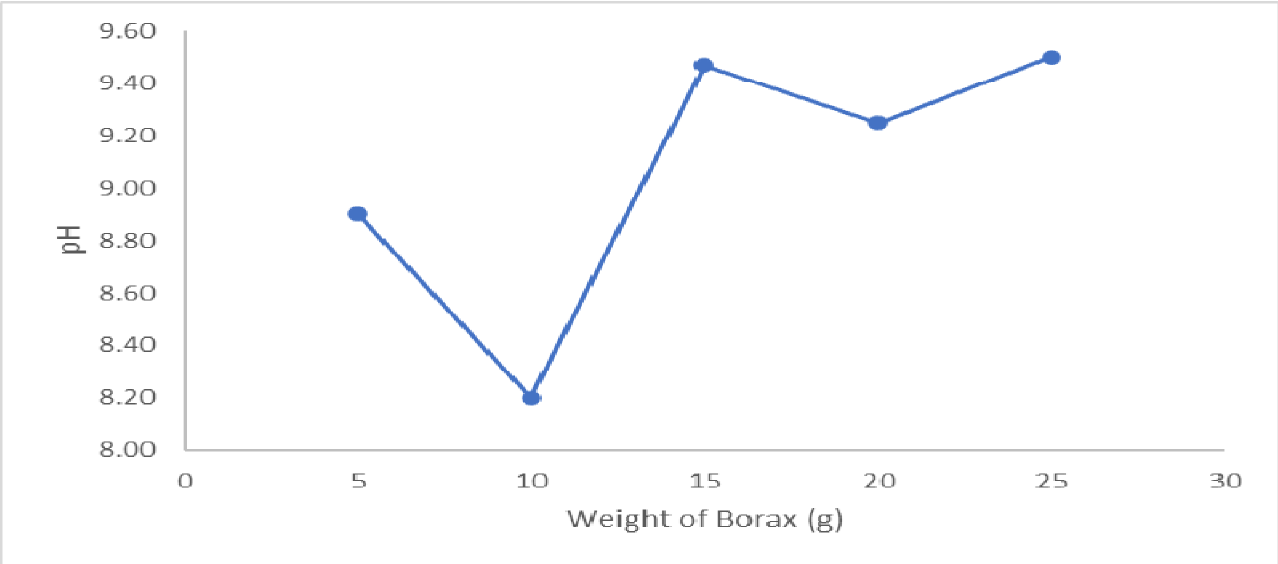

Fig 5: Plot of pH of starch-based adhesive to weight of borax

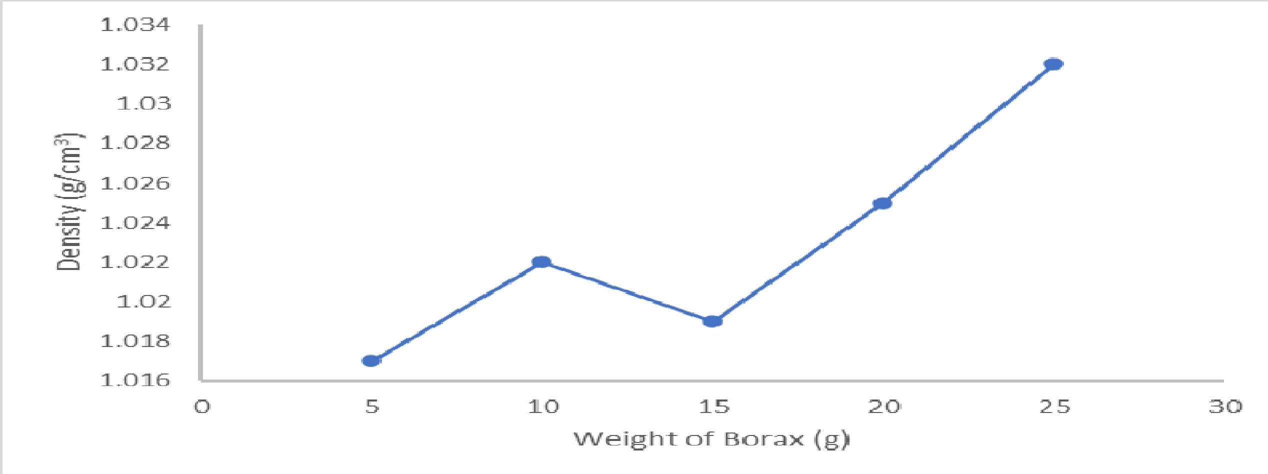

Fig 6: Plot of density of starch-based adhesive to weight of borax

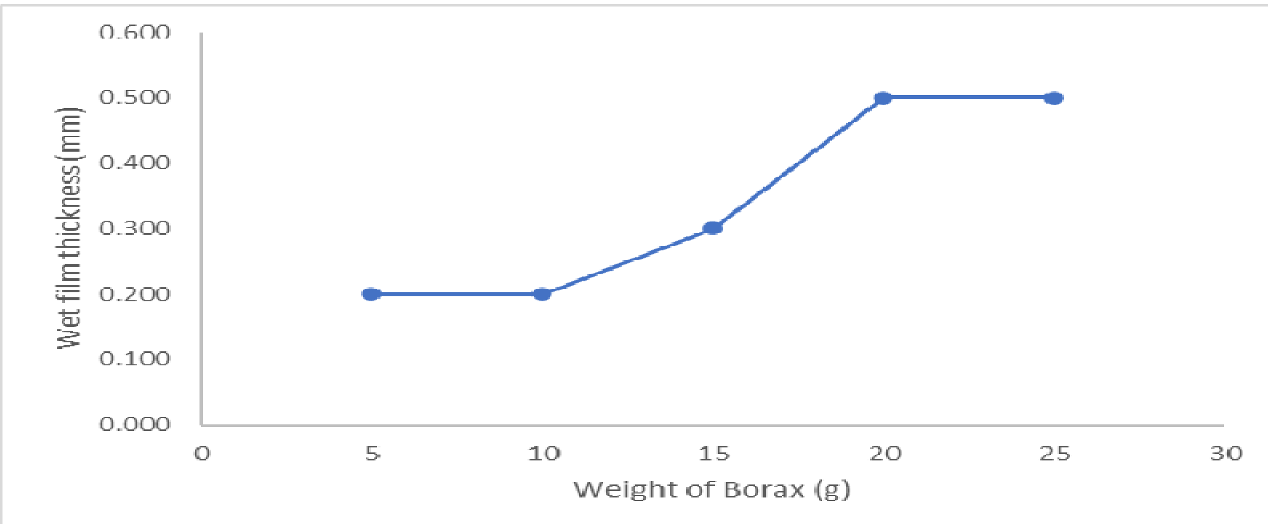

Fig 7: Plot of Wet film thickness of starch-based adhesive to weight of borax

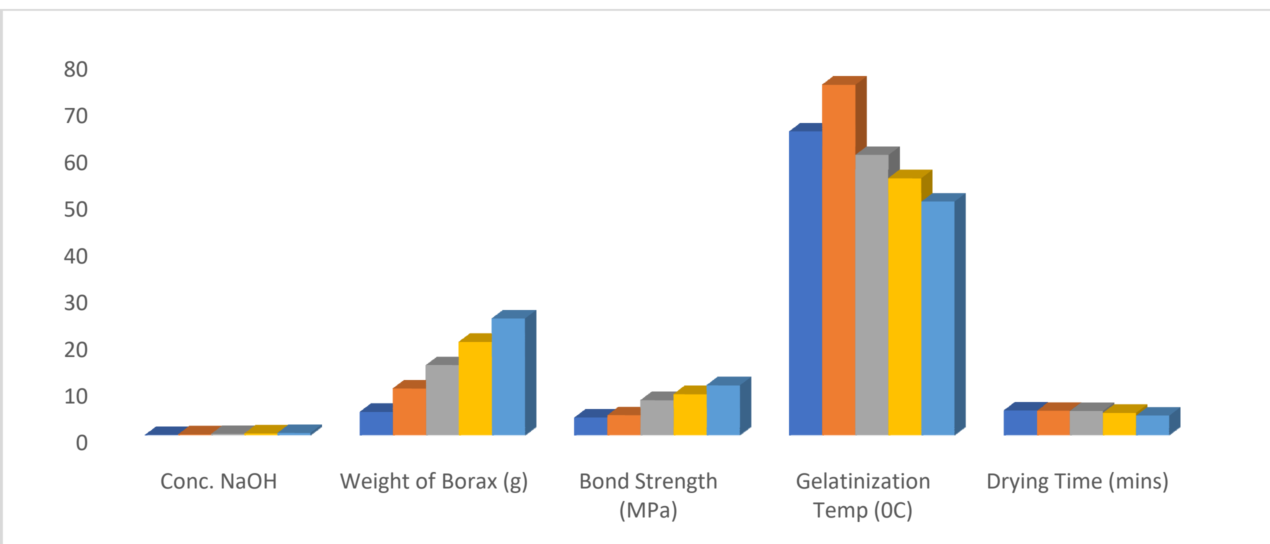

Fig 8: Bar chart illustrating the impact of Gelatinization modifiers and Viscosity enhancers on the adhesive characteristics of starch based adhesives

Sodium tetraborate (borax) plays a very critical role in the preparation of adhesives, it gives adhesives appropriate viscosities during application. The viscosity characteristics of adhesives are necessary in their rheological formulations hence the need for viscosity enhancers. An in-depth knowledge of the flow and deformation of the adhesive components is very important in understanding surfaces appropriate for specific adhesives, adhesives that must maintain their bond thickness during cure and adhesives that must obtain stability before full crystallization (Ojewumi et al., 2021). The process of gelatinization of starch is also essential in the adhesive characteristics of starch-based adhesive. Starch gelatinization is the subjection of starch to heat and water to obtain a viscous gel which can be applied as adhesives; however, this process can be modified with the introduction of an alkaline medium *((Aviara et al., 2010)*. Starch gelatinization is an endothermic process in which the crystallinity of the starch in its granules lost under specific moisture and heat. The viscosity of adhesives is very essential during the coating stage because it facilitates the curing process of the adhesive. Adhesives react differently when their molecules are yet to crystallize, they can only build up their final property and obtain their full strength after curing (Ubwa et al., 2012). Starch viscosity refers to its thickness, resistance to turbulence, shear or flow. The density of the adhesive refers to the molecular weight of the composition of the adhesive per unit volume. Both density and viscosity vary with temperature however their variations are not at the same rate while density has a linear variation with temperature, viscosity has a more rapid exponential variation with temperature (Dillard, 2011; Ojewumi et al., 2021). Comparison can be deduced that the rate of variation of the adhesive density with the gelatinization temperature is not rapid as observed with the viscosity. The wet film thickness is the measurement of the thickness of an applied adhesive before it dries up. It showed the impact of different weights of borax ($Na_2B_4O_7.10H_2O$) used as a viscosity enhancer as well as different concentrations of gelatinization modifiers (NaOH) on the physicochemical properties of starch-based adhesives. From the data, it can be deduced that the viscosity, density and wet film thickness of the adhesive increased with increase in the weight of the borax and concentration of the NaOH used respectively, there is however a consistency in the wet film thickness as the weight of borax and concentration of NaOH increased from 5.00g to 10.00g and 0.1M to 0.2M respectively. There is no specific trend in the pH of the adhesive while increasing the weight of borax and concentration of NaOH however the pH of the adhesives produced with the different quantities of viscosity enhancers (borax) and gelatinization modifiers are slightly alkaline as shown in the results obtained which is within specification. Increasing the pH of starch hydrolyses the starch molecules hence decreases its molecular weight resulting to low viscosity (Sadler and Murphy 2003). Plots of viscosity, pH, density and wet film thickness of starch-based adhesives versus weight of borax were recorded. Increasing the concentration of the gelatinization modifier (NaOH) and the weight of borax in starch adhesive increases the bond strength of the adhesive (Table 2). The bond strength of an adhesive is the adhesive's ability to hold itself together under stress. The higher the strength of cohesion within the molecules of the adhesive the stronger the bond strength of the adhesive and this is determined by the chemical composition of the adhesive. The acceptable bond strength for starch-based adhesives when dry is ≥ 7.3 MPa this infers that the addition of a gelatinization modifier (NaOH) with molar concentration of 0.3 M and a viscosity enhancer (borax) with a weight of 15 g to a starch adhesive can produce an adhesive with acceptable bond strength (Vanajasan et al., 2011). *T*he gelatinization temperature increased with increase in the concentration NaOH and weight of borax at concentrations of 0.1 to 0.2M for NaOH and 5 to 10g for borax. Above 0.2M NaOH and 10g borax it was observed that the gelatinization temperature reduced with increase in the concentration of the gelatinization modifier and weight of viscosity enhancers respectively. Gelatinization of starch can be defined as the temperature at which starch granules break open resulting in the release of amylose into liquid resulting in the thickening of the liquid (Ubwa et al., 2012). Gelatinization temperature of starch depends on plant type, pH, water and concentration of salt, sugar, fat and protein. Gelatinization of starch begins at 60 ^{0}C and the liquid fully thickens at 85^{0}C which is the gelatinization temperature (Aviara et al., 2010). The use of a gelatinization modifier helps in reducing the temperature at which the starch granules break open to release amylose which is essential in the conservation of energy. An initial increase in the gelatinization temperature at concentrations of 0.1 to 0.2M NaOH due to the strength of the straight chain α-1,4 glycosidic and branch chain α-1,6 glycosidic bonds which weakens with increase in the concentration of the gelatinization modifier (NaOH) (Dillard, 2011; Vanajasan et al., 2011). The drying time decreases with increase in the weight of borax and concentration of NaOH. Results obtained showed that the bond strength of the starch-based adhesive was inversely proportion to the drying time. Adhesives with better bond strength usually have less drying time compared to adhesives with relatively lower bond strength (Masumbu et al., 2003). It was showed that a bar chart illustrating the impact of gelatinization modifier (NaOH) and viscosity enhancer on the bond strength, gelatinization temperature and drying time of starch-based adhesive. The propensity of gel formation which is a function of the gelatinization temperature can be attributed to the viscosity of the adhesive and both characteristics are improved by the addition of gelatinization modifiers and viscosity enhancers.

Conclusion

Sodium tetraborate ($Na_2B_4O_7.10H_2O$) popularly known as borax and Sodium hydroxide (NaOH) can serve as viscosity enhancer and gelatinization modifier respectively when added at appropriate quantities to starch-based adhesives obtained from cassava (*Maniholt esculenta*) to produce vegetable glue with adequate bond strength. Physicochemical properties of starch-based adhesive from cassava (*Maniholt esculenta*) increased with increase in the quantities of added viscosity enhancer and gelatinization modifier. The gelatinization temperature of the starch-based adhesive reduced with increase in the concentration of the gelatinization modifier after experiencing an initial increase at lower concentrations due to the strong straight chain α-1,4 glycosidic and branch chain α-1,6 glycosidic bonds prevalent in cassava (*Maniholt esculenta*). The lower gelatinization temperature of starch-based adhesive from cassava (*Maniholt esculenta)* the easier release of amylose (polysaccharide of starch) from disintegration of starch granules and the higher bond strength of the adhesive. The higher viscosity of starch-based adhesive obtained from cassava (*Maniholt esculenta*) and better rheological formulations of the adhesive and this is directly proportional to the ability of adhesive to maintain stability of its molecular composition during curing (gel formation) which subsequently determines the bond strength of the adhesive.

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