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RESEARCH ARTICLE

FORMULATION OF CASSAVA STARCH-BASED ADHESIVE.

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Abstract

A more versatile and relatively cheap method of bonding virtually all materials together is by the use of adhesives. Among all the means of fastening, adhesive is the most suitable method because no damage is done to the adherends (as in nailing) and stress is more uniformly distributed. In this research work, various adhesives were produced from cassava which is a locally available natural raw material. The cassava starch was chemically modified to obtain oxidized, hydrolyzed and dexrinized from which the various adhesives were produced by incorporating other chemicals such as plasticizers and tackifiers. Generally, these adhesives were found have good bonding strength on wood, cardboard, paper and leather materials. However, adhesives produced from hydrolyzed and oxidized starch showed exceptionally good adhesive properties.

Introduction:-

An adhesive can be defined by the American Society for testing and Materials (ASTM) as a substance capable of holding materials together by surface attachment[1]. An adhesive must wet the surfaces, adhere to the surfaces, develop strength after it has been applied, and remain stable. Therefore adhesive is a generic term and covers other common terms, such as glue, paste, gums, adhesives, cement and bonding agent. A feature of adhesives is the relatively small quantities that are required compared to the weight of the final objects.

Adhesion as proposed by Wu refers to the state in which two dissimilar bodies are held together by intimate interfacial contact such that mechanical force or work can be transferred across the interface[2]. The interfacial forces holding the two phases together may arise from van der Waals forces, chemical bonding, or electrostatic attraction[3]. Adhesive resistance test results do not provide a direct reflection of a property of the material itself, but rather serve as an index of the behavior of the bonding system in the particular configuration of the test[4].

Starch is one of the most abundant natural, renewal biodegradable polymers and it is produced by many plants as source of energy.[5] Starch is made up of two molecules: amylose and amylopectin. Amylose consists of long helical chains, and amylopectin has a branched structure. It is extracted from several sources as semi-crystalline granules with different shapes and diameters. Starch is used and delivered most commonly as powder, in either bulk or bag form. Depending on the industrial application, it is either dissolved cold as it is or cooked[6]. The cassava starch has special technological properties that allow its utilization in many industrial applications. Among these properties are the absence of the typical “cereal flavor” of corn and other cereal starches, its ability of higher swelling degree

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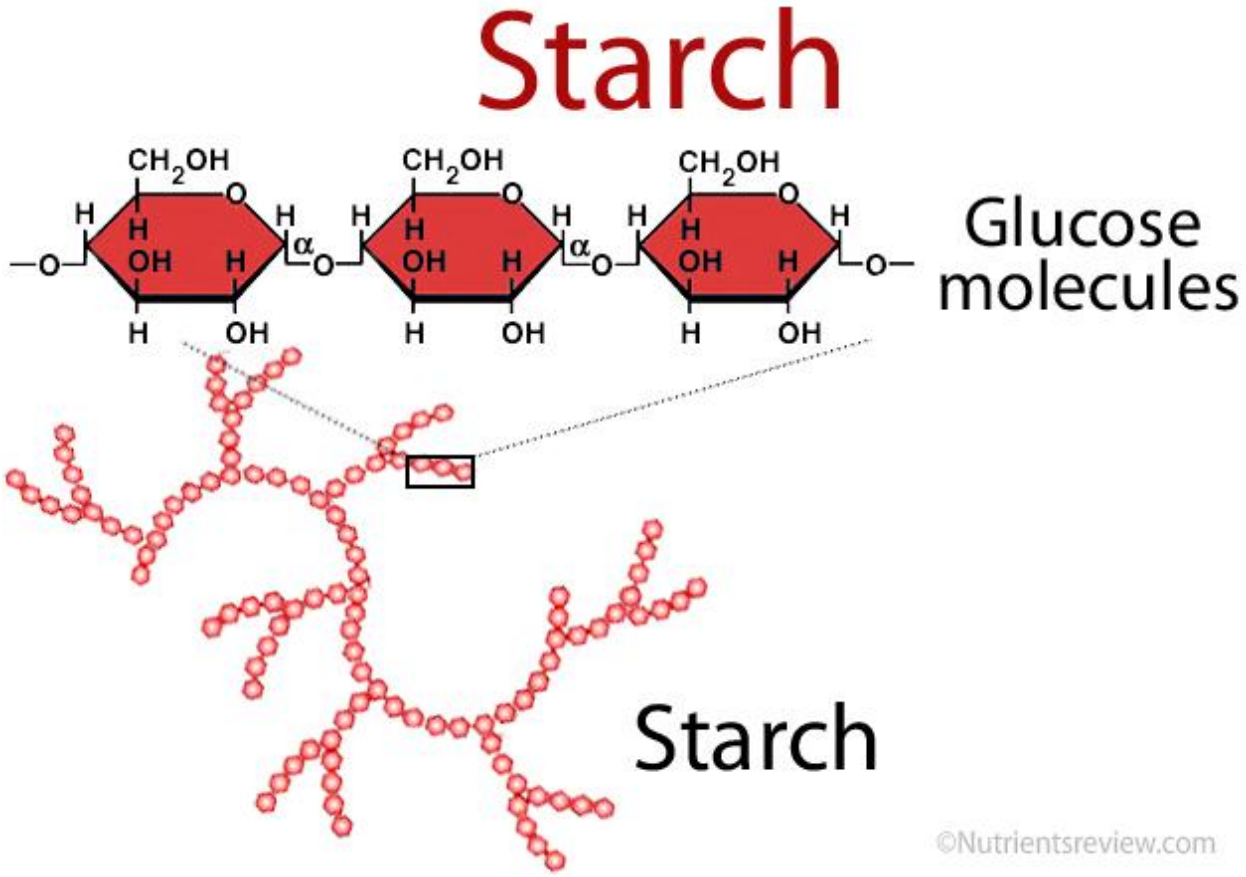
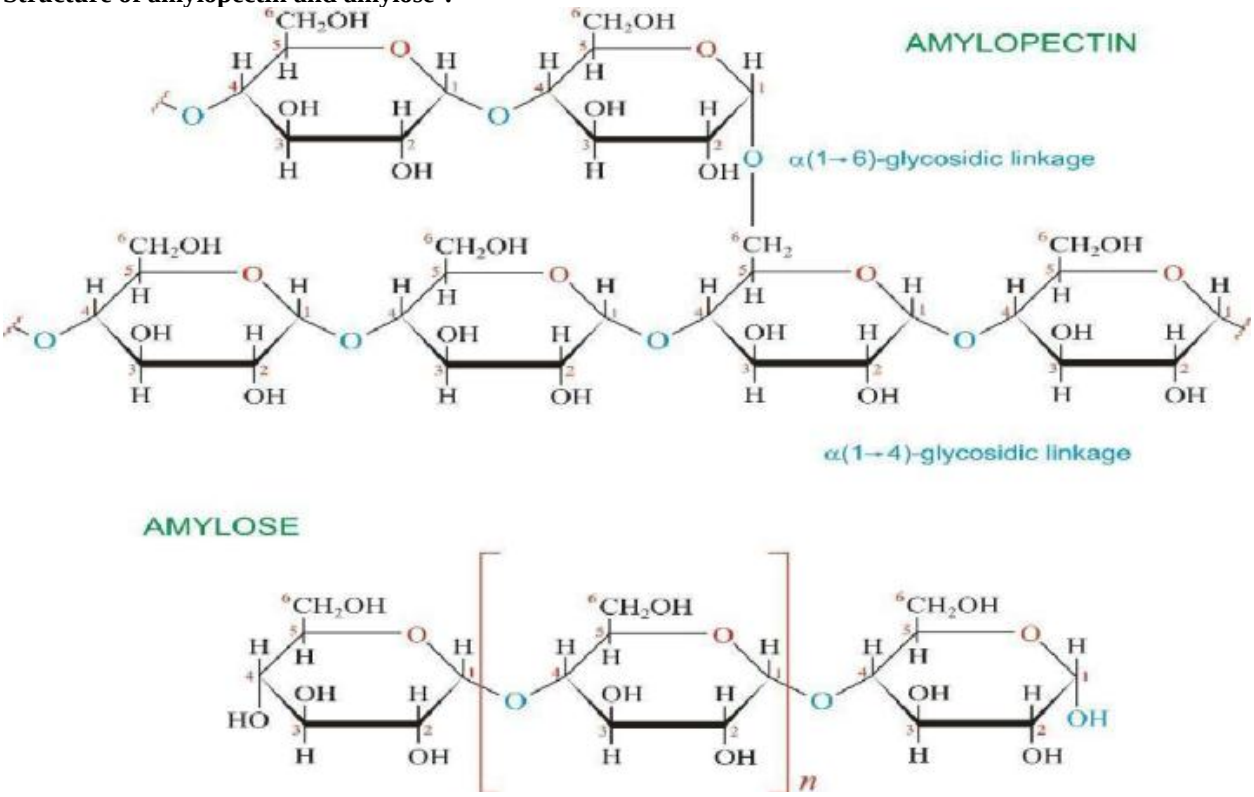
during cooking, and its lower pasting temperature, if compared again with cereal starches. Its low protein and lipid contents must also be valued contributing to its neutral flavor and white color[7]. Cassava is also cultivated in almost all parts of the country and it is quite cheap and readily available raw material for adhesive production. In compounding adhesive toluene which is petroleum solvent and some mineral acid are used.

Structure of Starch[8]:-



Structure of amylopectin and amylose[9]:-



The aim of this research is to formulate good quality adhesive that can bond paper, wood and also be used for bottle labeling using locally available raw material.

Modification for Starch adhesive production:-

Starch can be modified in order to improve viscosity and adhesion characteristics by a number of methods. They include;

a) By enzymes (maltose)
b) By acids (mineral)
c) By oxidizing agents (persulphates)
d) By heat

The industrial degradation products of starch may be broadly classified under the following groups;

1. Thin-boiling starches
2. Oxidized starches
3. Dextrin
4. Hydrolyzed

Materials and Method:-

Starch is principally a constituent of some kind of adhesive. It is abundant, cheap and simple in application. The starch used for this research was obtained from tuberous root of cassava plant in Abakaliki.

Preparation of cassava Starch:-

In preparation of cassava starch, the tubers were cut from the stems, washed, peeled and grated. This is normally done before 48 hours to prevent loss of starch due to enzymatic action and rot. The white mass were suspended in water and filtered with white sieve cloth. The filtrate was left to stand for 24 hours to ferment and then was filtered again and allowed to settle. It was then decanted and the white cassava starch was obtained. The white paste was dried to give a white powdery cassava starch.

Formulation of Adhesives from Modified Starch:-
Hydrolyzed Starch Adhesive:-
Caustic alkali is utilized in starch to produce an adhesive with good bonding strength.

Procedure:-
30 g of starch was suspended in 80 ml of water and the slurry stirred vigorously for about 30 minutes NaOH pellets (2g) were added while stirring the mixture until complete gelling of starch was obtained. This took about 10 minutes. HCl (8ml of 2 M) was then added to neutralize the starch. The resulting adhesive was extended with 5ml of glycerol and 2 ml of formalin was added as a preservative.

Dextrinized Starch Adhesive:-
Viscosity is reduced by use of dry heat, usually in the presence of acids. The process produces dextrin. This is the roasting of dry starch either alone or in the presence of an acid.

Procedure:-
40 g of starch was mixed with 5 ml of 0.1M HCl and then roasted for 30 minutes. The roasted starch was then ground into powdered form. This was then poured into 150 ml of warm water heated to 70^0C and thoroughly stirred with a mechanical stirrer. 2 ml of 2 M solution of caustic soda was added to increase the viscosity as well as neutralize the HCl. 5ml of formaldehyde was added as a preservative, 0.5 of borax was added to increase tack and the drying speed.

Oxidized Starch:-
The production of oxidized starch is similar to thin- boiling starch except that oxidation is done by oxidizing agents. Starch may be oxidized by many chemicals as sodium hypochlorite[7,10], bromine, potassium and ammonia persulfate, potassium permanganate, and H_2O_2 .

Formulation of paper adhesive from oxidized starch:-
60 g of starch was mixed with 2 g of $NaCO_3$ and 16 ml of H_2O_2 in a 500 ml beaker and the mixture stirred vigorously for 30 minutes to ensure proper oxidation of starch. 100 ml of water was then added to suspend the starch. 4 g of NaOH pellets was added stirring until complete gelling of starch occurred. 12 ml of 2M HCl was added. The adhesive obtained was extended with 10 ml glycerol and 4 ml formalin was added as a preservative.

Formulation of wood glue from oxidized starch:-
40 g of starch, 1 ml of H_2O_2, 0.5 g of sodium carbonate, 5 g of urea were mixed thoroughly in dry form and added gradually to 120 ml of water with constant stirring for 30 minutes using a mechanical stirrer. The mixture was heated to 70^0C, then caustic soda solution was added with constant stirring and heating until the adhesive had the desired consistency.

Results and Discussion:-

Starch constitutes a starting material to various adhesives ranging from paste to those involving complex formulation processes entailing several treatments. These adhesives formulated using starch were used for paper bonding, wood bonding, bag bonding and also in fast running machines for envelop making. Starch adhesives are quite cheap, readily available, and easy to apply and have measurable bonding strength that can withstand some environmental hazards.

Testing of Adhesives:-
Testing will be understood to mean the application of methods designed to determine whether the adhesive has been carefully produced, whether it will survive transportation and serve the purpose and most importantly whether it fits the requirement for which it was intended.

Test that are usually run on finished adhesives include solid content, viscosity, acidity or alkalinity, colour, odour and stability.

- Solid content: this is done using an infra-red lamp to drive off volatiles from the sample and the solid content weighed.
- Viscosity: viscometer is used for this purpose. Adhesive are non-Newtown liquids. Therefore the viscosity reading depends on the model of the instrument used.

- Alkalinity or Acidity: The pH meter is employed for this purpose
- Colour: variation in colour is usually not as important as variation in other properties. Nonetheless, for some user/uses, uniform colour affected by addition of desired pigments is a definite requirement.
- Odour: worthy of note as the fact that every adhesive has detectable odour when wet. Dry adhesives film usually has lower odour level than their wet components.
- Taste: this is usually important in envelopes since they are remoistened before use.
- Stability: the stability of industrial adhesive is usually not important since they are used immediately after production. But it is important for household adhesives since they can be stored for a long time.

Performance Test:-
The efficiency of adhesives which is relative can be predicted more accurately by measuring their drying speeds in industries.

The adhesive is applied and after a time of about 10-30 seconds, the surfaces are joined and the joints held under pressure for some time. This is referred to as ‘’temper time” and thus the strength of the bond is determined.

In the case of bottle labeling , the adhesive is cast on a glass plate and a sheet of paper cut into strips are torn out at 5 seconds interval until a fibre tearing bond is obtained.

There is also the case of carton sealing in which a film of adhesive is applied to cut strips of the carton to be joined. The film is allowed to temper for 30 seconds, after which the surfaces are joined together and pressure is applied for 20 seconds interval. The time at which a fibre tearing bond is obtained is noted. It is reasonable to assume at this point, that the carton flap would be bounded sufficiently to withstand open popping open unless some external pressure is applied.

Adhesive from the hydrolyzed Starch:-
A milky paste adhesive was obtained with pH of 11.0. This adhesive proved excellent in bonding of paper and for all kinds of paper labeling.
Paper to paper
Time taken to observe the fibre tearing bond is 60 s at temper time of 30s

Bottle labeling bonding
Time taken to observe the fibre tearing bond is 240 s at temper time of 60 s

Adhesive from oxidized starch:-
A milky paste adhesive was obtained with pH 11.2. This was used for book binding and labeling and it bounded quite strongly.
Paper to paper bonding
Time taken to observe the fibre tearing bond is 65 s, at temper time of 30 s
Bottle labeling bonding
The time taken to adhere the fibre tearing bond is 240 s at temper time of 60 s

Adhesive from dextrinized starch:-
A brown translucent adhesive with pH of 8 was obtained. The adhesive was used for bonding of paper and for bottle labeling and showed good bonding property.
Paper to paper bonding
Time taken to observe the fibre tearing bond is 75 s at temper time of 30 s

Bottle labeling bonding
Time taken to observe the fibre tearing bond is 245 s after temper time of 60 s

Wood Glue adhesive from oxidized starch:-
A yellowish brown paste adhesive with pH of 9 was obtained and it showed good bonding characteristics, this bonded strongly on wood. The time taken to separate the two pieces of wood joined by this adhesive after 60 s of application is 2 minutes. Therefore, 2 minutes is the separation time of this adhesive after 60 s of application. Wood adhesive used as standard is ‘Hard bond’ from market (trade name). The time taken to separate the two pieces of

wood joined by this adhesive after 60 s is also 2 minutes. Therefore separation time of this adhesive is equally 2 minutes.

Table 1:- Bonding Strength of the three Modified Cassava Based Starch on Paper at temper time of 30 seconds

Adhesive	Temper time (s)	Time taken to obtain the fiber tearing bond (bonding strength) (s)
Oxidized starch adhesive	30	65
Hydrolyzed starch adhesive	30	60
Dextrinized starch adhesive	30	75

Table 2:- Bonding Strength of the three Modified Cassava Based Starch on Bottle for Labeling at temper time of 30 seconds

Adhesive	Temper time (s)	Time taken to obtain the fiber tearing (bonding strength) (s)
Oxidized starch adhesive	30	240
Hydrolyzed starch adhesive	30	240
Dextrinized starch adhesive	30	245

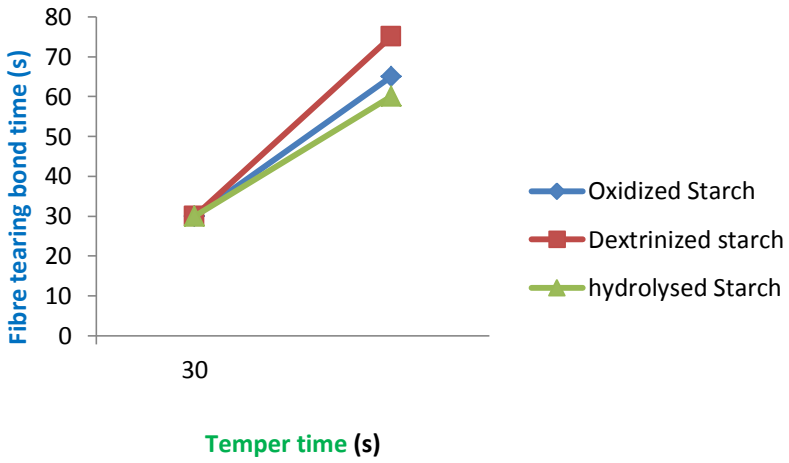


Figure 1:- Bonding Strength of three modified cassava starch adhesive on paper.

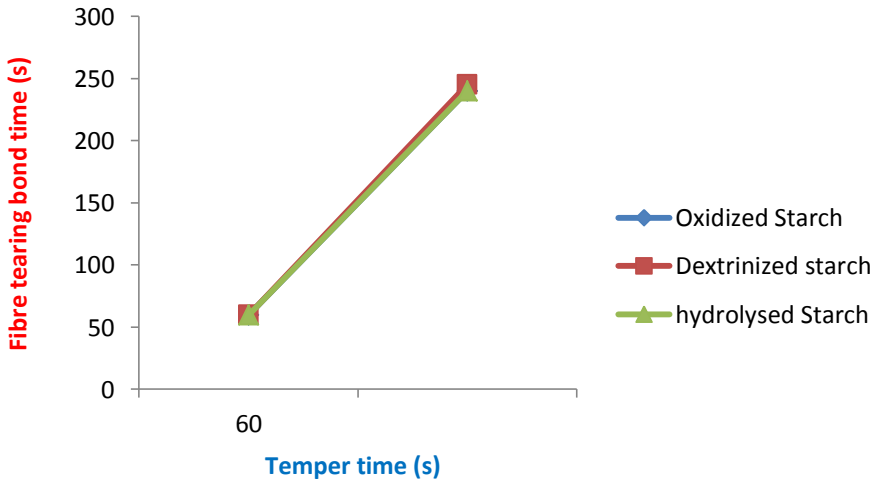


Figure 2:- Bonding strength of three modified cassava starch on bottle for labeling.

Bonding Strength:-
Generally, all the adhesives produced showed very good bonding strength on paper, wood, and for bottle labeling, but some showed exceptionally good bonding strength on paper and all kinds of bottle labeling. These include adhesive from hydrolyzed starch, oxidized starch adhesive. These adhesives can be used in place of synthetic glue, which was as standard. This synthetic adhesive could not bond very well on paper to bottle labeling. This may be due the fact that the adhesive is strictly for paper bonding. While the formulated adhesives were found to be equally good for bottle labeling. Also wood adhesive formulated actually showed very good bonding strength on wood, comparable to "hard glue" from the market, they have the same separation time which is 2 minutes.

Viscosity:-
Most of the adhesives produced retained their viscosity after one week of inspection, but two weeks after formulation, the dextrinized starch adhesive became slightly viscous. This could be attributed to the addition of borax which is a tackifier and consequently, increased the tackiness as well as the viscosity.

Drying time:-
This is a period of time during which an adhesive on adhered is allowed to dry with or without the application of heat or pressure. These adhesives formulated showed very short curing time of 60 s and 65 seconds respectively.

Colour:-
Most of the adhesives retained their colour after two weeks of formulation, but on second inspection after three weeks, oxidized adhesive changed from milky colour to pale ivory.

Stability:-
The formulated adhesive showed good stability after one week of formulation, but on the second inspection after three weeks, colour change was observed in oxidized starch adhesive, though there was no change in its bonding strength.

Cost Analysis:-
The cost analysis done for the formulated adhesives indicated that they are relatively cheap and affordable, thus commercial production of these starch-based adhesives will be a viable venture.

Conclusion:-

This research which is on formulation of adhesives from cassava starch has unfolded many facts relating to adhesive formulation. In the past, adhesives were made from animal skin, bones, gum Arabic and egg for veneering work. Recently, productions of adhesives are modified to involve other local raw materials, which are highly comparable with the synthetic types of adhesive. This is as a result of increase in the industrial use of adhesives more especially in shoe, wood and leather industries. This also can be attributed to advancement in technology.

From the performance tests and results, it has been demonstrated that good quality adhesives can be produced from cassava starch. The formulated adhesives have good bonding strength comparable to those in the market, good stability and also are cost effective. Finally, this research is quite interesting which one can venture into as a small-scale enterprise and become self-reliant.

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