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UTILIZATION OF CASSAVA FLOUR FOR PRODUCTION OF ADHESIVE FOR THE MANUFACTURE OF PAPERBOARDS

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INTRODUCTION

Cassava (*Manihot exculenta*) is a perennial vegetative propagated shrub mainly grown for its edible roots. The roots can be processed into major products as chips, flour, gari, starch, etc.

The composition of the roots varies according to such factors as age, variety, soil and climate. The starch content has been found to be as low as 12% and as high as 33%. The normal range at harvest time is from 22 to 31% (starch…) Moisture content varies directly with starch content on a weight basis, the normal content is between 60 and 75%. The rest of the root is cellulose (about 2%), proteins (3%), fats, mineral and soluble carbohydrates. Young roots, that is those less than 9 months old, are opt to be high in moisture (close to 70%) and low in starch (18-27%); starch content of old roots (over 24 months) is low, and are woody. In the manufacture of quality starch, the roots must be fresh.

The purified starch extracted from the minor root is commonly referred to in the trade, as "cassava flour" but cassava starch and cassava flour are the same. However, some producers refer to ground dried whole root as "cassava flour" but it is more properly cassava meal as it contains a considerable amount of fibre and root peel. The requirements of quality, quantity and end use of the cassava starch determine the processing technique to be employed in the manufacture. The application of starch in the food industry is widely known but other application in other industries is now coming up. Among the other application of starch are sizing paper and textile and in the alcohol. The industrial use of cassava starch is primarily determined by its physicochemical properties. In the adhesive industry wheat four and starch are used as extenders and fillers. Extenders are non-volatile paste-forming substances, which generally has some adhesive action either by modifying or enhancing the bonding effect of the resin adhesive mixtures. They are used mainly to improve perfomance and usually to reduce the amount of the primary bond required per unit area. The starchy and proteinous material improves spreading and setting behaviour of the adhesive.

Filler is a solid powder, non-volatile additive substance with little or no-paste forming effect. It is insoluble, inert cellulose made from wood waste or agricultural residue (Koch et al). It is dispersed in the resin mainly to help absorb the water released in the final curing reaction. In theory, fillers being finely particles present glue-line starvation i.e. controlling excessive adhesive penetration into porous wood.

Starch in the paper industry

The paper making process consists basically of preparing a fibrous form of cellulose and converting the fibres into a continuous web of paper. The cellulose fibre, that is, the basic raw material in the paper making operation would make a relatively low strength, poorly formed sheet if used unaltered in the paper machine. The fibre is, therefore mechanically refined to promote fibre-to-fibre bonding. There is, of course, a practical limit to improving their strength development beyond which the sheet loses desirable characteristics such as porosity, flexibility, brightness and opacity.
It is not always possible by refining alone to develop a finish that will produce an acceptable sheet. Often it is necessary to add binders to enhance the properties of the finished paper. Starch is the major binder used for this purpose.

Although starch may be used at the wet end of paper machine to compensate for paper refining or inadequate base fibre, its primary function is to increase the paper strength to lay surface fuzz and to increase stiffness and rattle or both. Use of starch also permits the inclusion for inorganic fillers without sacrificing the critical strength of the finished sheet. Many factors determine particular starch product is most effective in a given mill. These factors include the grades of paper being made, refining conditions and capacity, fibre type used, and economics.

The objective of this study, was to determine the varietal differences on the physiochemical properties of cassava flour and to know which of the varieties is best for the substitution of wheat flour in adhesive formulation for paperboard manufacture.

Experimental Methods and Materials

Source of material

The cassava tubers used in this study were obtained from Crops Research Institute (CRI) farm and Wenchi, Brong Ahafo.The varieties used were Abasafitaa (AB), Gblemoduade (GB) and Afisiafi (AF). All the varieties were aged 15 months.

The clay, which was used as an additive, was obtained from Afari in the Kumasi District. It was then washed with dilute HCl, dried, milled and sieved with a mesh of 0.07mm.Powdered rice glumes were obtained from CRI, Kumasi to be used an additive.

Characterization of cassava starch/flour

The parameters determined were:

1. Viscosity of starch

Fifty grams of the starch was weighed and dissolved in 500ml of distilled water. The suspension was stirred continuously and then placed on a hot plate with an electric regulator stirrer. When the solution got to about 65ºC temperature it was taken off the hot plate and cooled to room temperature. The viscosity was determined using spindle 3, as the speeds were changed in the increasing order and then decreasing order. Then at speed 6 rpm, with the spindle three; the viscosity was determined with change in time at intervals of 30 minutes.

2. Solubility

One gram of each starch sample was weighed and transferred into three different weighed graduated centrifuge tubes (50ml). Distilled water was added to each tube to give a total volume of 40ml.The suspension was stirred gently and uniformly, avoiding excessive speed since it might cause fragmentation of the starch granules. The samples in the tube were heated in a thermostatically regulated temperature bath for 30 minutes with constant stirring. It was removed, wiped dry on the outside and then centrifuged for 15minutes at 2200rpm. The supernatant liquor was then evaporated to dryness and the beaker weighed again to determine the solid residue. The percent solubility was calculated as in Appendix 1A.

3. Swelling Power

Here the process described in the determination of solubility was followed up to the point of 15 minutes centrifuge. The supernatant liquor was decanted and discarded. The tubes were then weighed to determine the sediment paste. The swelling power was then calculated as in Appendix 1B.

4. Water-Binding Capacity

Water binding capacity of starch was determined in triplicate according to the method of Yamazaki (1965). Two grams of starch was dissolved in 40ml of distilled water. The suspension formed was agitated for 1 hour on Griffin flask shaker (Griffin and George Limited, Great Britain). It was then centrifuged for 10 minutes at 2200 rpm. The free water

was decanted from the wet starch for ten minutes. The wet starch was weighed and the water binding capacity calculated as in Appendix 1C.

5. Percentage Amylose

The amylose content of the starch was determined based on the iodine colorimetric method described by McCready and Hassid (1943).

Preparation of Standard curve for Iodine colometric method and calculation of percentage amylose.

Different concentrations of pure amylose were prepare as follows: 10, 30,50, 70 and 90 mgs of pure corn amylose were weighed into separate 100ml volumetric flasks and wetted with ethanol (1 ml) and distilled water (10ml). The amylose was dissolved by adding 10% (NaOH) sodium hydroxide (2ml). The flask with its contents was cooled and diluted to the mark. A 5ml of this solution was poured into a 500ml volumetric flask. About 100ml of water was added and slightly acidified with 3 drops of 6M hydrochloric acid (HCl). The content was well mixed by shaking the flask and 5ml of iodine solution was added and diluted to the mark. The absorbance of each standard was read on a Cecil 8000 UV spectrophotometer at 640nm. Absorbance was plotted against percentage amylose. Linear regression analysis was carried out to derive an equation for determination of percentage amylose.

6. Preparation of Starch for Amylose Determination

100mg of the powdery starch was introduced into 100ml volumetric flask, wetted with 1ml of ethanol and 10ml of distilled water. The starch was dissolved by adding 2ml of 10% sodium hydroxide and heated on a water bath until a clearer solution was formed. The flask with its content was cooled and diluted to the mark. A 5ml portion of the alkaline starch solution was introduced into a 500ml volumetric flask and 100ml distilled water was added and slightly acidified with 34 drops of 6M hydrochloric acid (HCl). The contents were well mixed by shaking the flask and 5ml of iodine solution was added and diluted to the mark. The absorbance of the solution was read against a control that contains no starch in spectrophotometer at 640nm. The concentration of amylose was determined using equation derived from the standard curve as in Appendix 11.

7. Determination of pH

Two grams of starch was weighed and made into a slurry using 20ml of distilled water. The pH of the slurry was determined using a pH meter of model 526.

Adhesive Formulation

1. To prepare the starch adhesive without clay the following chemical were required in parts by weights.

Starch	-	100
Caustic soda	-	30
Sodium silicate	-	40
HCl	-	33
Formaldehyde	-	5
Borax	-	0.7
Water	-	500

Starch was suspended in water, sodium hydroxide was dissolved in water and stirred into it .HCl was added to neutralize the alkali partly and to it 150 parts more water was added. A secondary mixture was then prepared by dissolving sodium silicate in water, after which formaldehyde was added. The secondary mixture was then added to the adhesive and the mixture agitated for 15mm.

2. Preparation of adhesive with clay: A known weight of clay was dissolved in water to form slurry. Sodium silicate and formaldehyde were then added. The starch and clay slurries were then blended together in the correct proportion with constant stirring and the mixture heated to 60ºC.

The following chemicals were required in parts per weight:

Starch	-	100
Caustic soda (diluted with equal volume of water)	-	30
Sodium silicate	-	40
HCl	-	33
Formaldehyde	-	5
Borax	-	0.7
Water	-	500
Clay	-	1,2,3 & 4 gm

3. Starch adhesive preparation with rice glumes (husk) as additive: The following items (in parts by weight) were required to produce 500ml of starch adhesive:

Starch	-	36
NaOH	-	7
HCl	-	25
Formaldehyde	-	4
Rice husk	-	4,8,12 & 16

Tests and Analysis of Glue for Quality

Viscosity

To manufacturers and users, viscosity test is the most important in the evaluation of glue or gelatin. 500ml of the glue solution is measured into a 600ml beaker; it is placed in a water bath at 65ºC for 10 minutes. Then the viscosity determined with a Brookfield Synchro-Electric Viscometer of LV model.

ADHESIVE STRENGTH

A strip of 6cm wide of papers (machine glazed, craft paper, etc.) is marked of by folding leaving an area of 5cm square. 0.5ml of adhesive is evenly spread over the 5cm square cm area with a spatula. The paper pieces are stuck onto: -

a) A large piece of same paper
b) Cardboard

The specimens were kept for 4 hours at room temperature. The free 1cm wide flaps were then pulled with a steady pull.

KEEPING QUALITY

It is an index of the behaviour of the glue. Joiner's glue weighed into 200ml beaker containing 100ml cold distilled water. The content is kept for 16 hours at 10ºC with occasional stirring. The supernatant is then filtered with a strainer of stretch damped muslin of about 1/8cm mesh. The quality of water passing through the funnel in 5 minutes is measured. The difference between this water and 100 is the amount of water absorbed by the 10g of the glue.

CHEMICAL TEST

Solid Content

5.0g of the glue is weighed into a porcelain dish and evaporated to dryness on a water bath. The porcelain is transferred into an oven and maintained at 105 ± 2^{o} C for and hour. It is cooled and reweighed. Heating and cooling is done till a constant weight is obtained. This constant weight is the total solid content of 5.0g of the glue.

MOISTURE CONTENT

To test for this property, the glue sample was treated as described above (under solid content). The difference between 5.0g of the glue and the constant weight is obtained after heating gives the moisture content of the glue.

pH

The pH of each starch adhesive prepared was determined using a pH meter of model 526.

Table 1: Physiochemical properties of fresh cassava starch from three cassava varieties

Cassava Species	pH	Moisture Content (%)	Solubility (%)	Water Binding Capacity (%)	Amylose Content (%)	Swelling Power (%)	Viscosity Requirement (cp) Brookfield dial viscometer spindle/rpm
Abasafitaa	6.2	40.26	12.0	133.0	24.1	23.1	4,4000 LV 3/6
Afisiafi	6.4	44.7	7	144.0	21.6	22.8	7,750 LV 3/6
Gblemoduade	7.3	35.84	6.20	126.0	23.4	23.9	9,600 LV 3/6

Adhesive formulation from Abasafitaa Cassava species starch and (i) Clay and (ii) Rice glumes additives

Cassava	Additives		Viscosity Brookfield	Moisture Content (%)	pH	Solid Content	Water Absorption
	Clay (g)	Rice glumes (g)					
Abasafitaa	1	-	6,650 LV 3/6	15.8	6.8	67.62	27.50
"	2	-	7,600 LV 3/6	14.6	6.9	68.80	28.50
"	3	-	7,640 LV 3/6	12.4	6.9	69.61	28.50
"	4	-	9,810 LV 3/6	12.2	6.9	50.40	28.50
"	-	4	2,600 LV 3/6	13.5	6.9	52	26.4
"	-	6	2,800 LV 3/6	12.8	6.8	55	25.2
"	-	8	3200 LV 3/6	14.9	6.7	59	27.5
"	-	16	4000 LV 3/6	16.0	6.9	60	27.2

Discussion of Results.

CHARACTERIZATION PARAMETERS

Starch Yield

From the table, the starch yield of the three varieties fell between the range 17.33% and 28.56%. Gblemoduade had the lowest yield of 17.33% whiles the highest yield of 28.56% was Afisiafi and Abasafitaa gave the yield of 24.76%. These obtained values and the order in which they fell correspond to previous studies done on the same varieties (Barimah et al 1999). The amount of starch contained in root crops and tubers varies from 32% to 43% (Maud, 1990) and cassava roots contains 41% starch.

Moisture Content of Cassava Tuber

The moisture content of all varieties in this work was a bit low. The values from 35.84% for Gblemoduade through 40.26% for Abasafitaa to 44.71% for Afisiafi.These values obtained are below the literature stated value for cassava of 70% (Egglestone et, 1989). The moisture content of the cassava tuber decreases from time of uprooting thus a freshly cassava tuber would have a high moisture content than one which had been lying for some hours. The lower values obtained here would be due to different varieties used in both case studies.

pH

The standard pH specification for starch solution of 2% w/v is 4.5-7.0 (Sigma, 1999). The pH values of two of the three varieties fell within the range. The deviation of the obtained pHs from the upper limit of the standard pH range was approximately 0.3 units for Gblemoduade. Abasafitaa, which had the least pH, fell in the standard specified range, hence it preference to the other varieties. The pH of the starch solution was found not to vary significantly with changing concentration. This implies that more or less proton ions are not found in solution when the concentration is changed.

Solubility

Solubility values obtained were in the range 6%-12%. These values corresponded to similar work done on these same varieties and were in the same order (Barimah et al 1999). Gblemoduade had the least solubility of 6.29%, that of Afisiafi was 7% and Abasafitaa had the highest solubility value of 12%. The highest solubility of Abasafitaa goes to confirm its viscosity being the least at all speeds. The highest solubility means, the crystallinity and spatial structure of the starch polymer has been reduced (Alais et al, 1991). This means more sites of H-bonding available for interaction with water molecules. The higher the solubility value the more effective the starch would dissolve in the adhesive mixture and enhance its action.

Water-Binding Capacity

Water-binding capacity is the ability of starch granules to bind water molecules physically and chemically (Potter et al., 1995). Afisiafi had the highest WBC value of 144% followed by Abasafitaa with 133.0%. The least WBC value of 126% was for Gblemoduade. Abasafitaa was expected to have the highest WBC since from the solubility results Abasafitaa had its crystallinity and spatial structure broken. Faridi (1994) observed that when hard meet is milled, much of the starch is damaged. This is because during the milling a sizeable amount of shear stress is placed on the starch granules. The percentage of the starch granules that are subjected to shear stress of damage loose their order and crystallinity. When such granules are placed in water, they absorb much higher levels of water than undamaged granules. The Gblemoduade, which had the least solubility, had the least WBC, but far lower solubility and intermediate WBC value. This went to confirm Soni et al. (1987) that other factor that contributed to WBC are not only ultra-structure (molecular arrangement, amorphous and crystallinity areas) but also compositional (mainly amylose, amylopectin and phosphorus) characteristics of the starch and other factors. They reported that a loose association of amylose and amylopectin molecules in the native granules contributed to a high WBC. Hover and Sosulki (1986) reported that engagement of hydroxyl groups to form Hydrogen and covalent bonds between starch chains might lower WBC. Wooton and Bamunuarachi (1978) also reported that differences in WBC of starches result from different degrees of availability of water –binding sites to the hydroxyl groups and interglucose oxygen atom. During gelatinisation of starch, the water-binding sites are increased due to interruption of the inter-granular bonds by heat.

Swelling Power

Swelling power gives an idea of how much water is able to enter into the amyloplast of starch granules. Kordylas (1990) reported that starch granules or units begins to absorb water and to swell even when temperature reaches about 20º to 30º C. Gblemoduade had the highest value of 23.88%, which was followed by Abasafitaa with 23.06%. Afisiafi had the least value of 22.7%. Swelling power value thus has a range of 2 approximately 1.34. This means that there is no significant difference between the varieties in their ability to absorb water into their amyloplast.

Percentage Amylose

The amylose content results obtained ranged from 21.6 and 23.4 percent for Abasafitaa, Afisiafi and Gblemoduade respectively. The results obtained in this work were far higher than some reported values of amylose content of cassava starch. For the same cassava species and in the same order. Barimah et al (1999) reported values were 24.6,25.4,27.10 percents. Also Rickard et al. (1991) values ranged from 13.6% to 23.8%. Another set of values of amylose content of cassava starch ranged from 22.6% to 26.25%(Monthly et al., (1992).

Viscosity

The viscosity was measured with spindle three at increasing and decreasing speeds and at a temperature of 30ºC. It was observed that viscosity does not change with passage of time. This confirmed the fact that starch is a Non-Newtonian fluid. The least viscosity for Abasafitaa means more of the glucosidic linkages have been broken. This provides more

bonding sites for enhancing gluing nature of the adhesive. The highest viscosity of Afisiafi was to five the highest swelling power but that was not observed.

CONCLUSION

From the characterisation of the three varieties, Abasafitaa was chosen for the formulation of the adhesive. Its characteristic of having more amylose than the others implies more cross-linking in adhesive mixture. Abasafitaa had the highest solubility that made it the best to dissolve readily in the adhesive glue mix. Abasafitaa's pH was the least and nearest to the recommended pH range of values. The swelling power and WBC of Abasafitaa were intermediate, making them absorb water and hold water at the glue-line for transfer and flow. Abasafitaa will resist the strong absorptive forces of paper thus more effective in glue mix than other starches.

Also from the results of adhesive the formulation 2g, of the clay was adequate for the improvement of the gluing characteristics of the starch adhesive. It's solid –content and moisture, which were intermediate, allows it to spread and cover more area, set faster, has high tenacity and can stay longer on the shelf.

APPENDIX I

A.

Calculation of % solubility

% Solubility= (wt. of soluble starch * 100) /wt. of sample (dry basis)

B.

Calculation of swelling power

Swelling power = (wt. of sediment paste* 100) /[wt. of sample (dry basis)*(100- % solubility)]

C.

Calculation of % WBC

% WBC = (bound water *100) / wt. of sample (dry basis)

D.

Equation for % amylose

Equation derived from linear regression analysis for % amylose

$Y = 29.35x – 0.0091$

Y = absorbance (nm)

X = concentration of amylose (g/l)

APPENDIX II

Standard curve for Iodine Colometric method

Absorbance vrs. Conc. Of amylose

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