# Optimization of White Dextrin for the Production of Starch Based Adhesive

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**Abstract**
This research studies the optimization of white dextrin for the production of starch based adhesive using cassava starch. It involves the optimization of the three process variables namely, roasting temperature, roasting time and concentration of acid using Minitab 17 design of experiment in order to obtain the setting that gives the best solubility for white dextrin. A range of 70 to 90°C, 0.3 to 0.35 M and 30 to 90 minutes were used for temperature, concentration and time respectively. The result obtained shows that roasting temperature of 90°C, concentration 0.35 M and roasting time of 30 minutes gave the best solubility for white dextrin (32.6 %). Factorial analysis was used to determine the individual and combined effects (interaction) of the process parameters on white dextrin solubility. In addition an empirical model was developed for the factorial analysis. Result from this experiment shows that the process variables (roasting temperature, roasting time and concentration of acid) have significant effects on the solubility of the dextrin produced. Factorial analysis further suggested that a white dextrin of best solubility is produced at roasting temperature of 90°C, concentration of 0.35 M and roasting time of 30 minutes. It gave a model equation for the predicted value of white dextrin solubility as 32%.

**Keywords:** Adhesive, Starch, Dextrin, Solubility, Design.

## Introduction

Starch is a food crop which is very important to man. About 60-70 % of calories taken by humans is gotten from starch (Lawton, 2004). Apart from it been nutritive it is used as raw material in food, pharmaceutical, paper, construction industries. According to Burrell (2003) non-food application of starch includes adhesives in packaging and paper industry, match header in explosives, pill coating and dispersing agent in pharmaceuticals. This application depends on its functional properties such as pasting, gelatinization and solubility which vary from one botanical source to another with environmental condition and variety (Yuan et al., 2007). Therefore the use of starch for both food and non-food industries calls for a better understanding of their unique functional, physicochemical and structural properties. The most common source of starch include maize, wheat, potato, cassava, rice (Vaclavik and Christian, 2008).

Cassava is one of the most important starchy root crop in the world that is used for both food and industrial purposes, it is use in the manufacture of garri, starch flour and other products in Nigeria, it is also used to produce adhesive. Cassava is of the family Euphorbiaceae and genius manihot it has become a food crop of high potential and industrial value, it was introduced into Nigeria by early Portuguese trader Buba and Kabiru

(2014). In most manufacturing industries like packaging, paper, brewing, and food processing industries adhesives are indispensable materials.

Adhesives are polymer substance capable of binding materials together. It is a semi-synthetic low molecular carbohydrate which is formed as a result of hydrolysis of starch such as cassava (tapioca), potatoes, rice and corn (Akpa, 2012). It is produced under acidic condition such as pyrolysis and roasting which is made by mixing starch with a particular concentration of hydrochloric acid (HCl) or nitric acid ($HNO_3$) and heat is applied. For an adhesive to be effective, it should be able to impact adequate bond between materials and it should be easy to apply as well as have resistance to moisture as observed by Ubadike (2015). There are hundreds of adhesives in the world market today each varying in material of production and formulation. Dextrin is normally in white, yellow and brown colour which are fully soluble at times and partially insoluble most times. The smooth texture, non-poisonous and non-staining nature of starch makes cassava adhesives a desirable choice for domestic use. However numerous problems are involved with the use of starch adhesives such as very short shelf life.

Design of experiment is a series of test in which usefully changes is made to the input variables of a process and the effect are measured. Montgomery (2005) stated that necessary approach to process and product design and development which involve design of experiment consist of three phases: characterization, control and optimization. This research focuses on production of adhesive from cassava starch. The optimization of dextrin will first be carried out by varying the process variables which are roasting time, roasting temperature and concentration of acid. The response that will be used in the optimization process is the solubility of dextrin.

## Materials and Methods

*Materials, Chemicals and Equipment*

Cassava starch used for this experiment was obtained from Minna, Niger State, Nigeria. The chemicals/Reagent used are: Distilled water, hydrochloric acid (HCL), sodium hydroxide (NaOH), borax ($Na_2B_4O_7.10H_20$) and formal dehyde ($CH_2O$). the equipment/Glassware used are: Stop watch, viscometer, electric oven, plastic funnel, thermometer, mesh sieve (250 µm), spatula, magnetic stirrer with hot plate, centrifuge, pH meter, digital weighing balance, 250 ml volumetric flask, 50 and 100 ml Measuring cylinders and 250 ml beakers.

*Experimental procedure.*

Already processed cassava starch was purchased from Capital market Minna, Niger State, Nigeria and was sieved using a 250 µm mesh sieve. Starch base adhesive process was developed using a $2^3$ experimental design and the variables considered were concentration of acid, roasting time and roasting temperature. The factorial design software chosen for the analysis was Minitab 17 software and sixteen test were made. Each of the design was made randomly in other to distribute the errors.

In preparing dextrin from cassava starch the method used was by Gumus and Udezue (2011) with slight modification. 25 g of dried cassava starch was mixed with 200 ml 0f 0.35 M HCl (gelatinization enhancer) and aged for 24 hr. At a temperature of 90°C the mixture was heated using a heater with magnetic stirrer for 30 minute. These steps was repeated for all other experiment but with variation in the variables.

Dextrin solubility was determined using the method describe by Aytunga et al., (2010) as earlier used by Leach et al. (1959) with little modifications. 2.5 g of the prepared dextrin was dissolved in 30 ml of distilled water and was heated for 15 min using hot plate maintained at 50°C. It was allowed to cool and then centrifuged at 2000 rpm for 10 minute. The supernatant was separated and heated for 15 min and the solubility was measured as mass of supernatant over mass of heated supernatant multiply by 100 %.

In preparing adhesive for paper bag, 178.8 g of white dextrin having 32.1 % solubility in water was mixed with 16 g borax, 0.04 g formaldehyde and 0.12 g of liquid paraffin the mixture was heated at 85°C for 30 minute. And cooled to room temperature and 4.8 g of 50 % NaOH was added to the mixture.

The pH of the samples were determine using digital pH meter. 20 ml of each sample was put into a sample bottle and the pH was determine by inserting the pH meter electrode into it. The values was gotten from the display of the pH meter.

The viscosity was determined at room temperature using Brookfield viscometer set at a speed of 60 rpm with spindle number 29 and expressed in pa.s.

The drying time of the adhesive which is the time it takes for the adhesive to set after been covered with substrate was determined manually, using a stop watch

**Results and Discussion**

The solubility of dextrin is the evidence of water molecules and starch chain interaction, it increases with increase in heating (temperature) as the temperature increases water penetrates more into the amorphous region of the starch granules therefore resulting to more hydration and dissolution due to the swelling of the granules (Peroni et al., 2006).

White dextrin is prepared at low temperature, in the presence of large amount of acid and a shorter time duration. It is usually white in colour and slightly soluble in water. The temperature range of 70 to 90°C was used for the production of white dextrin as the solubility that will be obtained at a temperature lower than that will be unsuitable. Table1 shows the temperature, acid concentration (0.3 to 0.35 M) and roasting time (30 to 90 minutes) range which was used to produce white dextrin at these ranges it was seen that the conversion of starch was slow and gives a white colour while an increase in the temperature gives a more brownish colour and solubility range of 27.0 to 32.6 % was obtained this is due to the different rate at which the bursting of the starch granules occur.

Table 1 shows that moderate temperature of 90°C and acid concentration of 0.35 M and roasting time of 30 minutes gives the best solubility of 32.6 %.

**Table 1:** Solubility (Response) at Different Roasting Temperatures, Time and Concentrations for White Dextrin

| Run order | Temperature (°C) | Roasting Time (min) | Acid Concentration (M) | Solubility (%) |
|---|---|---|---|---|
| 1 | 90 | 30 | 0.35 | 32.6 |
| 2 | 70 | 30 | 0.30 | 27.7 |
| 3 | 90 | 30 | 0.30 | 28.9 |
| 4 | 70 | 30 | 0.35 | 27.0 |
| 5 | 90 | 90 | 0.35 | 28.9 |
| 6 | 90 | 90 | 0.30 | 30.9 |
| 7 | 70 | 90 | 0.35 | 28.5 |
| 8 | 70 | 90 | 0.30 | 28.1 |
| 9 | 90 | 90 | 0.35 | 28.6 |
| 10 | 70 | 90 | 0.30 | 27.6 |
| 11 | 90 | 30 | 0.35 | 32.1 |
| 12 | 70 | 90 | 0.35 | 28.0 |
| 13 | 90 | 30 | 0.30 | 28.5 |
| 14 | 90 | 90 | 0.30 | 30.4 |
| 15 | 70 | 30 | 0.30 | 27.3 |
| 16 | 70 | 30 | 0.35 | 27.5 |

Table 2 shows the estimated coefficient and effects for the solubility of white dextrin. It shows that the model contains three main effect which are significant since there p value is less than 0.05, Table 2 shows the p-value result which indicate that there is a significant interaction between temperature and time (0.002) and also between temperature and concentration (0.038) since their p-values is less than 0.05 ($\alpha = 0.05$). Also from the table roasting temperature and concentration of acid has a positive effect which means that an increase in this variables will result to an increase in the solubility of white dextrin and an increase in roasting time which has a negative effect will result to a decrease in the solubility of white dextrin. It also shows that roasting temperature has the greatest effect (2.4000) on dextrin solubility when compared to other factors and setting a high temperature will give higher solubility than a low temperature. The interaction between temperature, time and concentration gives the next greatest interaction (-1.5500) on solubility the negative sign shows the setting of the three process variables have contending effect.

The interaction between roasting time and concentration of acid has the next greatest effect (-1.2250) on dextrin solubility. In addition, it shows that roasting temperature is set low and concentration of acid is set high to produce dextrin of higher solubility. Roasting time has the smallest effect (-0.0750) on the solubility of dextrin setting a low time will give a high solubility. The order of the effects and statistical significance of the formulation is shown in a Pareto chart as shown in Figure 1. Table 2 shows the estimated effects and coefficients for solubility of white dextrin.

**Table 2** Estimated Effects and Coefficients for Solubility of White Dextrin

| Term | Effect | Coeff | SE Coe | T-Value | P-Value |
|---|---|---|---|---|---|
| Constant | | 28.9125 | 0.0805 | 359.05 | 0.000 |
| Temperature | 2.4000 | 1.2000 | 0.0805 | 14.90 | 0.000 |
| Time | -0.0750 | -0.0375 | 0.0805 | -0.47 | 0.654 |
| Concentration | 0.4750 | 0.2375 | 0.0805 | 2.95 | 0.018 |
| Temperature * Time | -0.75 | -0.3750 | 0.0805 | -4.66 | 0.002 |
| Temperature * Concentration | 0.4000 | 0.2000 | 0.0805 | 2.48 | 0.038 |
| Time * Concentration | -1.2250 | -0.6125 | 0.0805 | -7.61 | 0.000 |
| Temperature * Time * Concentration | -1.5500 | -0.775 | 0.0805 | -9.62 | 0.000 |

Pareto chart of the standardized effect for white dextrin solubility response of Figure 1 is a bar graph that shows information in order of magnitude to graphically show the relative importance of the difference between groups of data. The red line represent 95 % confident level it shows that there are three significant effect ($\alpha$=0.05), which include roasting temperature (A) roasting time (B) and concentration of acid

(C). Pareto plot shows that largest effect is roasting temperature because it extends the farthest and the smallest effect is the roasting time because it extends least.

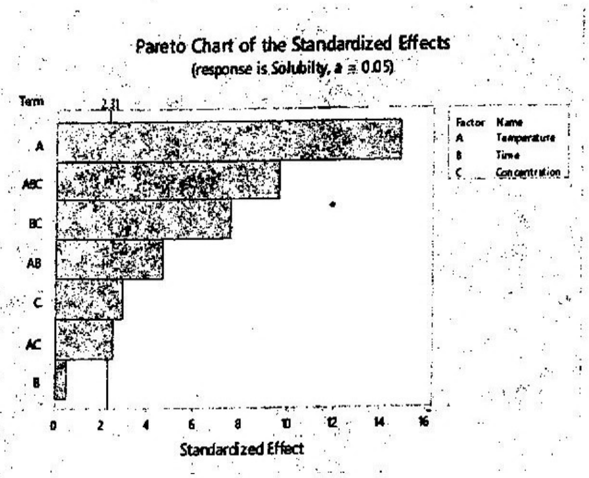


**Figure 1:** Pareto Chart of the Standardized Effect for White Dextrin Solubility

Normal plot of standardized effect for white dextrin solubility response in Figure 2 shows there are also three significant effect (α= 0.05). These are the three main effects namely, roasting temperature (A) roasting time (B) and concentration of acid (C). Roasting temperature has the largest positive effect because it lies furthest from the line. In addition the plot indicates the direction of the effect, concentration of acid have a positive effect since it reside to the right of the line. This means that when the process variable changes from high to low the solubility of dextrin reduces. The significant of the process factors and their rating are in agreement with the estimated result of the effect and coefficients for the solubility in Table 2. Figure 2 shows the normal plot of the standardized effect for white dextrin solubility.

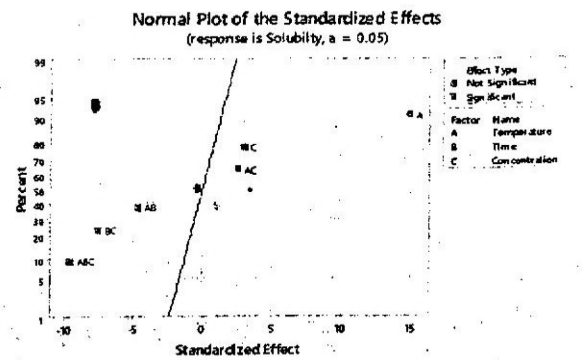


**Figure 2:** Normal plot of the Standardized Effect for White Dextrin Solubility

Figure 3 shows the half normal probability plot for white dextrin solubility. Which also shows that there are three significant effect: roasting temperature (A) roasting time (B) and concentration of acid (C) with roasting temperature having the highest effect because it lies furthest from the line.

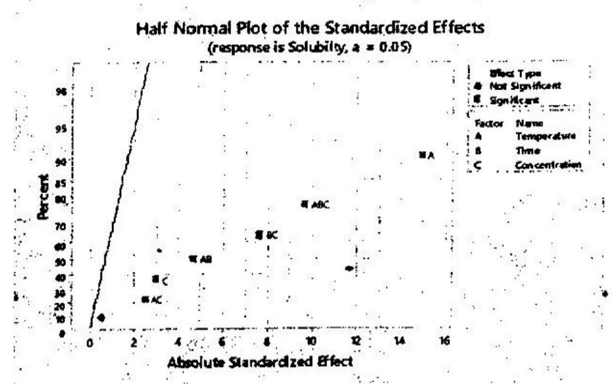


**Figure 3:** Half Normal Plot of the Standardized Effect for White Dextrin

The cube plot of Figure 4 illustrate that if high concentration of acid, low roasting time and high roasting temperature are used, the solubility of white dextrin is 32.35 %

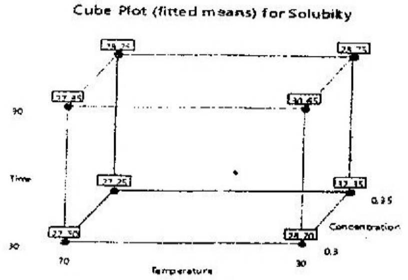


**Figure 4** Cube Plot for White Dextrin Solubility

The model equation for white dextrin solubility which is built from Table 2 is shown in the Equation below.

$$Y_w = 28.9125 + 1.2000x_1 + 0.2375x_3 - 0.3750x_1x_2 + 0.2000x_1x_3 - 0.6125x_2x_3 - 0.7750x_1x_2x_3$$

$Y_w$: The predicted value of white dextrin solubility

$X_1$: The roasting temperature which has two levels coded 1 and -1

$X_2$: The roasting time which has two levels coded 1 and -1

$X_3$: The concentration of acid which has two levels coded 1 and -1

The model equation via the combination above of the main effect and the interaction plot is:

$$Y_w = 28.9125 + 1.2000(1) + 0.2375(1) - 0.3750(1)(-1) + 0.2000(1)(1) - 0.6125(-1)(1) - 0.7750(1)(-1)(1)$$

$Y_w = 32.3125$.

## Conclusions

The optimum condition for the production of white dextrin was obtained at temperature of 90°C, roasting time of 30 minutes and concentration of 0.35 M which gave a solubility of 32.6 %.

From the factorial analysis roasting temperature roasting time and concentration has significant effect on the solubility of dextrin. The analysis also suggested that there are significant interactions between the individual variables to produce dextrin of high solubility.

White dextrin was used to produce paper bag adhesive with a viscosity of 3 pa.s, drying time of 3.05 minutes and pH of 7.64.

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