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ADHESIVE FROM GUYANA CASSAVA STARCH

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ABSTRACT. Appliying statistical design techniques, good adhesives from cassava starch were obtained by the acid hydrolysis at 96 to 98 °C. The most significant variable in the range that was studied was the ratio of water to starch which in general should be 2,5 and could be 1,5 if it is used the higher acid concentration (1 %) and time (10 min) to obtain a semiliquid. Employing the relation 2,5 considering the higher concentration (1 %) it was obtained a semiliquid with the shortest time (5 min) but a liquid with 10 min. Also with the ratio 2,5 it was possible to obtain a good paste employing the smaller acid concentration (0,5 %) but the longer time (10 min).

RESUMEN. Mediante la aplicación de técnicas de diseño estadístico se logró obtener buenos adhesivos mediante la hidrólisis ácida de 96 a 98 °C de almidón de yuca de Guyana. La variable más importante, en el rango estudiado, fue la relación de agua a almidón que en general debe tomar el valor de 2,5, pero puede emplearse 1,5 si se toma la mayor concentración de ácido (1 %) y tiempo (10 min) para obtener un líquido muy viscoso. Utilizando la relación 2,5 y considerando la mayor concentración (1 %) se obtuvo un líquido muy viscoso en 5 min y otro bastante fluido en 10 min. También fue posible con la relación 2,5 obtener pastas blancas usando la menor concentración de ácido (0,5 %) y los tiempos mayores (10 min).

INTRODUCTION

Starch has been obtained, probably, for the first time from corn, and has been used as food by the Romans and Greeks[1]. Starch is found in corn, cassava, potatoes, sweet potatoes, wheat, etcétera[1].

Starch represented by $C_6H_{10}O_5$ is constituted by two polymers, one linear (amylose) and another branched (amylopectin)[1-4].

Starch is employed in great amounts in the Food, Paper, Textiles and Adhesive Industries. Its employ should increase with the general decline in the utilization of petroleum[4-8].

In this paper it was studied the acid hydrolysis of cassava starch to obtain dextrins in solution which enabled us to develop adhesives for different purposes as for example; white adhesives for printing, offices, labels, paper bags, etcétera.

EXPERIMENTAL METHOD

The acid hydrolysis of cassava starch was studied following the techniques developed by López Planes and col.[9,10] employing hydrocloric acid (HCl).

A 2^4 factorial design was chosen to perform the experiments considering four independent variables[11].

X_1 acid concentration (0,5 % and 1 %)

X_2 time of reaction at 96 to 98 °C (5 to 10 min)

X_3 ratio of water to starch (1,5; 2,5)

X_4 borax content (5 and 10 % on starch basis)

The matrix D of experiments shows which experiments should be done:

$$
\begin{array}{ll}
 & \begin{array}{lll} X_1 \text{ (acid concentration)} & X_2 \text{ (time)} & X_3 \left(\frac{\text{water}}{\text{starch}}\right) \end{array} \\
\underline{D} = & \left|\begin{array}{lll}
-1 \ (0{,}5\ \%) & -1 & -1 \\
1 \ (1\ \%) & -1 \ (5\text{ min}) & -1 \\
-1 & 1 \ (10\text{ min}) & -1 \\
1 & 1 & -1 \ (1{,}5) \\
-1 & -1 & 1 \ (2{,}5) \\
1 & -1 & 1 \\
-1 & 1 & 1 \\
1 & 1 & 1 \\
0 \ (0{,}75\ \%) & 0 \ (7{,}5\text{ min}) & 0 \ (2{,}0)
\end{array}\right|
\end{array}
$$

At the bottom if this matrix is also shown the centre experiments that were performed.

In order to save space it was wrote only the 2^3 factorial design for the first three independent variables (X_1, X_2, X_3). The fourth independent variable, X_4, was considered by doing the above experiments with 5 % borax, then adding the required amounts of borax to half of the adhesive produced form each experiment, to make 10 %. That meant that 16 experiments plus four centre experiments were performed.

With these results it was should be able to determine all the coefficients of the following polynomial:

$$
\begin{aligned}
Y = {} & b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_{12}X_1X_2 + \\
& + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{23}X_2X_3 + b_{24}X_2X_4 + \\
& + b_{34}X_3X_4 + b_{123}X_1X_2X_3 + b_{124}X_1X_2X_4 + \\
& + b_{134}X_1X_3X_4 + b_{234}X_2X_3X_4 + b_{1234}X_1X_2X_3X_4
\end{aligned}
$$

by means of the well known regression matrix[11]

$$B = [\underline{X}'\ \underline{X}]^{-1}\ \underline{X}'\underline{Y}$$

where:

$\underline{B}$ vector of coefficients =

$= [b_0, b_1, b_2, b_3, b_4 \ldots b_{1234}]^1$

$\underline{X}$ matrix of the independent variables

$\underline{X}'$ transpose of the independent variables

$\underline{Y}$ vector of the results or yields

The selected results or yields were the so–called time for tackiness, measured as the time in which a piece of paper of 3 cm long by 1 cm wide adhered to another similar piece, spreading the adhesive on a 1 mm portion. The time in which the adhesive was just able to sustain a 400 g weight was measured.

It was also made tearing test, which was simply to tear the portion where the two pieces of paper stock together to determine whether the adhesive separated easily or the paper was broken. The later case was described as a positive test which meant that the adhesive really the two pieces of paper together.

Some important qualitative properties were also noted. For example, the physical state at room temperature of the adhesive. When a solid was obtained, it was determined whether a gel was obtained or not. It a gel was obtained then the adhesive was considered unsuitable. If the adhesive adhered to the finger when touched it was considered suitable for mechanical spreading.

RESULTS

The experimental results are shown on Table I.

Taking the time for tackiness as the yield the 16 coefficients of the polinomial were determined; obtaining:

$$Y = 125 - 34X_1 - 28X_2 - 82X_3 + 24X_4 + 20X_1X_2 + 31X_1X_3 - 8X_1X_4 + 31X_2X_3 + 21X_2X_4 - 24X_3X_4 + 22X_1X_2X_3 - 30X_1X_2X_3 + 14X_1X_3X_4 - 9X_2X_3X_4 - 8X_1X_2X_3X_4$$

The variance of the pure error was determined by the usual equation for 2 repetitions per experiment, obtaining[11]:

$$S^2_{pe} = 2\ 306$$

$$S_{pe} = 48$$

The 95 % confidence interval for each coefficient should be:

$$2Sb_i = 2\sqrt{S^2_{pe}/16} = 24$$

Considering this, the polinomial should be written as:

$$Y = 125 - 34X_1 - 28X_2 - 82X_3 + 31X_1X_3 + 31X_2X_3 - 30X_1X_2X_3$$

First of all it was noticed that the variables $X_1 X_2$ and X_3 should be increased in order to diminish the time for tackiness, X_3 (ratio between water and starch) was the most important variable. That mean that the ratio 2,5 should be used and this should permit us to obtain a higher volume of adhesive per 100 g of starch. In the second place, time and acid concentration should be increased. It is very important to notice also that the X_4 variable does not appear in the polinomial that means that the borax concentration is not significant in which case we should use the lower concentration (5 %). Besides it is observed from Table I that the experiment 10 (10 % borax) behaves worst than experiment number 2 (5 % borax). The same happens for numbers 3 and 11.

TABLE I

Experiment	Time for tackiness (s)	Tearing Test	Physical State	Remarks
1 (5 % borax)	240 to 420	no good	solid, gel	Does not adhere to the finger. Difficult to spread
9 (10 % borax)	240; 300; 360	no good	solid, gel	Does not adhere to the finger. Very difficult to spread
2 (5 % borax)	120 to 210	positive	semisolid paste	Cream colour. Adheres to finger better than number 10
10 (10 % borax)	240 to 300	positive	semisolid paste (almost a gel)	Cream colour. Adheres not so good to finger. Tend to form balls
3 (5 % borax)	60 to 90	positive	semisolid paste	Cream colour. Adheres to finger
11 (10 % borax)	270 to 300	positive	solid, gel	Cream colour. Does not adhere to finger
4 (5 % borax)	60 to 75	positive	semiliquid	White colour. Adheres very good to finger. Very good
12 (10 % borax)	60 to 75	positive	semisolid paste	White-cream colour. Adheres very good to finger. Very good
5	75 to 90	positive	solid, gel	White-cream colour. Does not adhere to finger well
13	30 to 45	positive	solid, gel	White-cream colour. Does not adhere to finger, difficult to spread
6	15 to 30	positive	semiliquid	White colour. Very good. Adheres very good to finger.
14	15 to 20	positive	semiliquid	White colour. Very good. Adheres very good to finger
7	15 to 30	positive	semisolid paste	White colour. Very good. Adheres very good to finger
15	40 to 45	positive	semisolid paste	White colour. Very good. Adheres very good to finger
8	30 to 60	positive	liquid	White. Adheres very good to finger. Very good
16	30 to 60	positive	liquid	White. Adheres very good to finger. Very good
C-2 (5 % borax)	30 to 45	positive	semisolid paste	Dark-cream colour. Adheres to finger well
C-3 (10 % borax)	120 to 150	positive	solid, gel	Dark-cream colour. Adheres to finger not so well
C-4 (10 % borax)	150 to 180	positive	solid, gel	Dark-cream colour. Adheres to finger not so well
C-5 (5 % borax)	30 to 60	positive	semisolid paste	Cream. Adheres to finger

Considering this and the general results, four adhesives are recommended for different purposes.

Numbers 4 and 6: suitable for book binding, labels, offices, paper bags, etcétera.

Number 7: suitable for book binding, offices, boxes nom–water resistant, schools

Number 8: suitable for book binding, and offices (where a liquid id preferred).

Applying statistical design techniques, good adhesive from cassava starch were obtained by the acid hydrolysis at 96 to 98 °C.

The most significant variable in the range that was studied was the ratio of water to starch which in general should be 2,5 and could be 1,5 if we use the higher acid concentration (1 %) and time (10 min) to obtain a semiliquid (experiment 4).

Employing the relation 2,5 considering the higher acid concentration (1 %) it was obtained a semiliquid with the shortest time (5 min) but a liquid with 10 min (point 6 and 8).

Also with the ratio 2,5 it was possible to obtain a good paste employing the smaller acid concentration (0,5 %) but the longer time, 10 min (experiment number 7).

The results also showed that it was better to use the smaller borax concentration (5 %). Without any doub the results showed that cheap adhesives for different purposes can be obtained from cassava starch.

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