Novelty Journals

ISSN 2394-7349

International Journal of Novel Research in Engineering and Science
Vol. 12, Issue 1, pp: (20-29), Month: March 2025 - August 2025, Available at: www.noveltyjournals.com

Optimization of Process Parameters for Yellow Dextrin Production

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DOI: https://doi.org/10.5281/zenodo.15370781

Published Date: 09-May-2025

Abstract: **Starch was processed from freshly harvested cassava, dried and stored for dextrin production. The starch was mixed separately with HCl and HNO_3(nitric acid) as catalysts at different concentrations, temperatures and times. This was to determine the effect of the process parameters on the production of yellow dextrin. Temperature range of 50 to 90°C was adopted while acid concentration range of 0.1 to 0.5 M and time range of 20 to 60 minutes were used for the dextrinization process. The process parameters for the dextrinization of starch was optimized using Central Composite Design (CCD) expert Stat-Easy 360. The results obtained showed that the temperature of 80°C and roasting time of 30 minutes with concentration of 0.4 M using HCl gave a dextrin with the highest solubility of 76.596%, while using Nitric acid, temperature of 80°C, roasting time of 30 minutes and concentration of 0.4, M gave a Dextrin with the highest solubility of 53.6172%.**

Keywords: **Adhesive, Cassava, Catalyst, Dextrin, Response surface method, Starch.**

I. INTRODUCTION

Starch is a natural polymer which is made from mixture of two distinct polysaccharide fractions of amylose and amylopectin, which both are made out of glucose of various sizes and shapes [1]. Starch molecules put together by each plant species have distinct structures and compositions, and the protein and fat content of the storage organs might differ significantly. This natural functional difference enlarges its range of industrial uses[2] Starch is a vegetable substance made up of amylose, which is a linear chain consisting of about five hundred to two thousand units, and amylopectin, which is highly branched and consist of over one million glucose units[3]. It is a complex carbohydrate that exists in many foods, including grains, vegetables, and fruits. The principal sources of starch are cassava, maize, tapioca, wheat, potatoes. The chemical reactivity of starch is dependent on the response of the constituent glucose units [4]. The chemical and functional properties achieved following such modification depends largely on the reaction conditions such as modifying reagent(s), concentration of the reactants, reaction time, type of catalyst used, pH, and temperature. It is also used as raw material in food processing, pharmaceutical, paper industries, cosmetics match header in explosives, pill coating and dispersing agent in pharmaceuticals [5,6,7,8].

The cassava roots are rich in starch and approximately half of the total roots made are procesed for starch production. Although corn has been the first crop for starch production (more than 80% of the world starch production), cassava production for starch is increasing speedily (over 3% annually) accounting for about 7% of global starch made[9]. Cassava starch has numerous advantages compared to other crops such as higher starch accumulation capacity, all the year-round availability, economical price, resilience to drought, poor soil factors, pests and diseases, and relatively simple starch extraction method [10]. Cassava starch can be produced from either wet or dry milling [11].

Dextrins are starch hydrolysis products made by acid hydrolysis, enzyme hydrolysis or a combination of both [12]. Dextrins can be made from any starch and are generally classified as white dextrins, yellow dextrins, and British gums. It is more water-soluble and produces less viscous solutions or dispersions than its parent starch. Each is produced by combinations of breaking down starch and and molecular rearrangement. Dextrins are used in the manufacturing of adhesives for case and carton sealing, in food, textile and cosmetics industries[13]. Dextrins are made by dry (filtration) or wet methods. Dry method (filtration) causes color change in starch during the process. Therefore, the dextrin produced has darker color. Hence dry method was used to produce dextrin with the addition of acid of enzymes. Dextrin made with acid catalyst has an advantage as the process is easy, material is easy to get and cheap[14]. Whereas dextrin made with enzyme can be done in a non-extreme process [15]. Dextrin is polydextrose in pharmaceutical fields that work as diluents for pills and capsules, binder, sugar coating material as plasticizer, adhesive and thickening agent for suspension [16].

Adhesive are polymer substance that can join materials together by a process of adhesion[17]. It is a semi-synthetic low molecular weight carbohydrate formed by the hydrolysis of starch that can be obtained from any of cassava, potatoes, rice and corn. It can also be produced by pyrolysis or roasting after mixing starch with quantified amount of hydrochloric acid (HCl) or nitric acid (HNO_3). It involves interaction of the adhesive surface with the substrate surface being held in close contact such that mechanical force or work can be transferred across the interface[18]

Previous studies were done on Preparation and Characterization of Soluble Dextrin Fibre from Potato Starch Obtained on a Semi-Industrial Scale [19], Production of Adhesive from Cassava Starch[20], Cassava starch-based hot melt adhesive [21], and Optimization of biodegradable starch adhesives using response surface methodology [22].

The aim of the study is o optimize the effect of process parameters for yellow dextrin production

1) To process starch from freshly harvested cassava

2) To produce dextrin from the starch using HCL and Nitric acid as catalysts.

II. METHODOLOGY

Cassava were peeled, washed and grated to finer particles. The starch was then extracted from the grated pulp by sieving while the fiber was retained. The fiber retained was washed repeatedly for at least three (3) to four (4) times with distilled water on the screen. The starch extracted was allowed to sediment after which the fiber was decanted off and the starch is rewashed with distilled water to remove the remaining fiber. The starch was then dried in an oven at a temperature of (45^{o}C) for six (6) hours to reduce the amount of moisture content and finally dried under a brilliant sunshine for four (4) hours. The powdery starch was then stored in an air tight container to prevent contamination and moisture.

Design Matrix For Dextrin Production From Cassava Starch

The process factors for dextrinization of starch was optimized using Central Composite Design (CCD) expert Stat-Easy 360. The 25g starch was mixed separately with HCl and HNO_3 as catalysts at different concentrations, temperatures and times. This was to determine the effect of the process parameters on the production of dextrin. Temperature range of 50 to 90°C was adopted while acid concentration range of 0.1 to 0.5 M and time range of 20 to 60 minutes were used for the dextrinization process. Table I showed the design matrix for dextrin production with 40runs.

TABLE I: DESIGN MATRIX FOR DEXTRIN PRODUCTION

Std Order	Run Order	Acid Conc (M)	Time (Mins)	Temp (°C)	Type of acid
19	1	0.3	40	70	HCL
5	2	0.2	30	80	HCL
34	3	0.3	40	90	Nitric acid
6	4	0.4	30	80	HCL
22	5	0.4	30	60	Nitric acid
26	6	0.4	30	80	Nitric acid
23	7	0.2	50	60	Nitric acid
25	8	0.2	30	80	Nitric acid

16	9	0.3	40	70	HCL
27	10	0.2	50	80	Nitric acid
18	11	0.3	40	70	HCL
8	12	0.4	50	80	HCL
31	13	0.3	20	70	Nitric acid
35	14	0.3	40	70	Nitric acid
32	15	0.3	60	70	Nitric acid
12	16	0.3	60	70	HCL
1	17	0.2	30	60	HCL
29	18	0.1	40	70	Nitric acid
7	19	0.2	50	80	HCL
20	20	0.3	40	70	HCL
11	21	0.3	20	70	HCL
33	22	0.3	40	50	Nitric acid
9	23	0.1	40	70	HCL
13	24	0.3	40	50	HCL
21	25	0.2	30	60	Nitric acid
24	26	0.4	50	60	Nitric acid
36	27	0.3	40	70	Nitric acid
2	28	0.4	30	60	HCL
38	29	0.3	40	70	Nitric acid
28	30	0.4	50	80	Nitric acid
40	31	0.3	40	70	Nitric acid
3	32	0.2	50	60	HCL
37	33	0.3	40	70	Nitric acid
17	34	0.3	40	70	HCL
10	35	0.5	40	70	HCL
39	36	0.3	40	70	Nitric acid
14	37	0.3	40	90	HCL
30	38	0.5	40	70	Nitric acid
4	39	0.4	50	60	HCL
15	40	0.3	40	70	HCL

III. RESULTS AND DISCUSSION

The results of design matrix for dextrin production were shown in Table II. It showed the percentage solubility of dextrin. It showed the results for 40 runs in different concentrations of acid(0.1 to 0.5M), roasting times (20 to 60minutes) and temperatures of 50 to 90^{O}C.

TABLE II: RESULTS OF DESIGN MATRIX

Std Order	Run Order	Acid Conc (M)	Time (Mins)	Temp (°C)	Type of acid	Solubility of Dextrin (%)
19	1	0.3	40	70	HCl	73.8423
5	2	0.2	30	80	HCl	69.2718
34	3	0.3	40	90	Nitric acid	49.7133
6	4	0.4	30	80	HCl	76.596
22	5	0.4	30	60	Nitric acid	46.0223
26	6	0.4	30	80	Nitric acid	53.6172
23	7	0.2	50	60	Nitric acid	41.3158

25	8	0.2	30	80	Nitric acid	48.4903
16	9	0.3	40	70	HCl	73.8114
27	10	0.2	50	80	Nitric acid	49.55548
18	11	0.3	40	70	HCl	73.078
8	12	0.4	50	80	HCl	75.816
31	13	0.3	20	70	Nitric acid	48.9926
35	14	0.3	40	70	Nitric acid	51.72258
32	15	0.3	60	70	Nitric acid	50.4231
12	16	0.3	60	70	HCl	72.033
1	17	0.2	30	60	HCl	57.6186
29	18	0.1	40	70	Nitric acid	40.5787
7	19	0.2	50	80	HCl	70.7928
20	20	0.3	40	70	HCl	73.8894
11	21	0.3	20	70	HCl	69.9894
33	22	0.3	40	50	Nitric acid	39.8307
9	23	0.1	40	70	HCl	57.9696
13	24	0.3	40	50	HCl	54.821
21	25	0.2	30	60	Nitric acid	40.333
24	26	0.4	50	60	Nitric acid	48.8288
36	27	0.3	40	70	Nitric acid	51.68982
2	28	0.4	30	60	HCl	65.7462
38	29	0.3	40	70	Nitric acid	51.10014
28	30	0.4	50	80	Nitric acid	53.0712
40	31	0.3	40	70	Nitric acid	51.15474
3	32	0.2	50	60	HCl	59.0247
37	33	0.3	40	70	Nitric acid	49.5277
17	34	0.3	40	70	HCl	73.0002
10	35	0.5	40	70	HCl	72.4308
39	36	0.3	40	70	Nitric acid	51.668
14	37	0.3	40	90	HCl	71.019
30	38	0.5	40	70	Nitric acid	50.70156
4	39	0.4	50	60	HCl	69.7554
15	40	0.3	40	70	HCl	70.7538

The percentage solubility for nitric acid showed the lowest value at 0.3M concentration, 40minutes and 50^{O}C was 39.8307% and highest value at 0.4M concentration, 50minutes and 80^{O}C was 53.0912%. The percentage solubility for HCl showed the lowest value at 0.3M concentration, 40minutes and 50^{O}C was 54.821% and highest value at 0.4M concentration, 30minutes and 80^{O}C was 76.596%. Hence percentage solubility of HCL gave the highest value of 76.596%. It was in line with what observed by [23],

ANALYSIS OF VARIANCE

TABLE III: ANOVA TABLE

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	5389.32	10	538.93	371.61	< 0.0001	significant
A-Acid Conc	326.52	1	326.52	225.14	< 0.0001	
B-Time	9.48	1	9.48	6.53	0.0161	
C-Temp	455.47	1	455.47	314.06	< 0.0001	

D-Chemical Type	4262.62	1	4262.62	2939.19	< 0.0001	
AC	7.66	1	7.66	5.28	0.0290	
AD	10.17	1	10.17	7.01	0.0130	
CD	19.11	1	19.11	13.18	0.0011	
A^2	141.38	1	141.38	97.49	< 0.0001	
B^2	9.82	1	9.82	6.77	0.0144	
C^2	215.53	1	215.53	148.62	< 0.0001	
Residual	42.06	29	1.45			
Lack of Fit	31.35	19	1.65	1.54	0.2446	not significant
Pure Error	10.71	10	1.07			
Cor Total	5431.38	39				

Std. Dev.	1.20	R^2	0.9923
Mean	58.74	**Adjusted R^2**	0.9896
C.V. %	2.05	**Predicted R^2**	0.9828
		Adeq Precision	61.5874

The **Model F-value** of 371.61 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise.**P-values** less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AC, AD, CD, A^2, B^2, C^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.The **Lack of Fit F-value** of 1.54 implies the Lack of Fit is not significant relative to the pure error. There is a 24.46% chance that a Lack of Fit F-value this large could occur due to noise [24], [25]. The **Predicted R^2** of 0.9828 is in reasonable agreement with the **Adjusted R^2** of 0.9896; i.e. the difference is less than 0.2.**Adeq Precision** measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 61.587 indicates an adequate signal. This model can be used to navigate the design space.

MODEL EQUATIONS

Chemical type: HCL

Dextrin solubility = -110.99232 + 186.62030 Acid Conc + 0.407963 Time + 3.56056 Temp – 0.691911 Acid Conc*Temp – 167.67741 Acid $conc^2$ – 0.004419 $Time^2$ – 0.020703 $Temp^2$ (1)

Chemical type: Nitric Acid

Dextrin solubility = -117.43782 + 175.34688 Acid Conc +0.407963 Time + 3.40601 Temp -0.691911 Acid Conc*Temp – 167.67741 Acid $Conc^2$ – 0.004419 $Time^2$ – 0.020703 $Temp^2$ (2)

The equation in terms of actual factors can be used to make predictions about the response for given levels of each factor. Here, the levels should be specified in the original units for each factor. This equation should not be used to determine the relative impact of each factor because the coefficients are scaled to accommodate the units of each factor and the intercept is not at the center of the design space.

MODEL GRAPHS

Interaction Plot of Temperature and Acid Concentration: This is the 3-D interaction plot. As the temperature and the acid concentration were increased the solubility value also increased until a maximum was obtained at the peak of the graph. Further increase in the factors started showing decrease in the solubility value. This could be attributed to how temperature and acid concentration jointly affect dextrin solubility, depending on whether the lines are parallel, converging, diverging or crossing, you can infer whether there is no interaction, a positive or a negative interaction between these factors.

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ISSN 2394-7349

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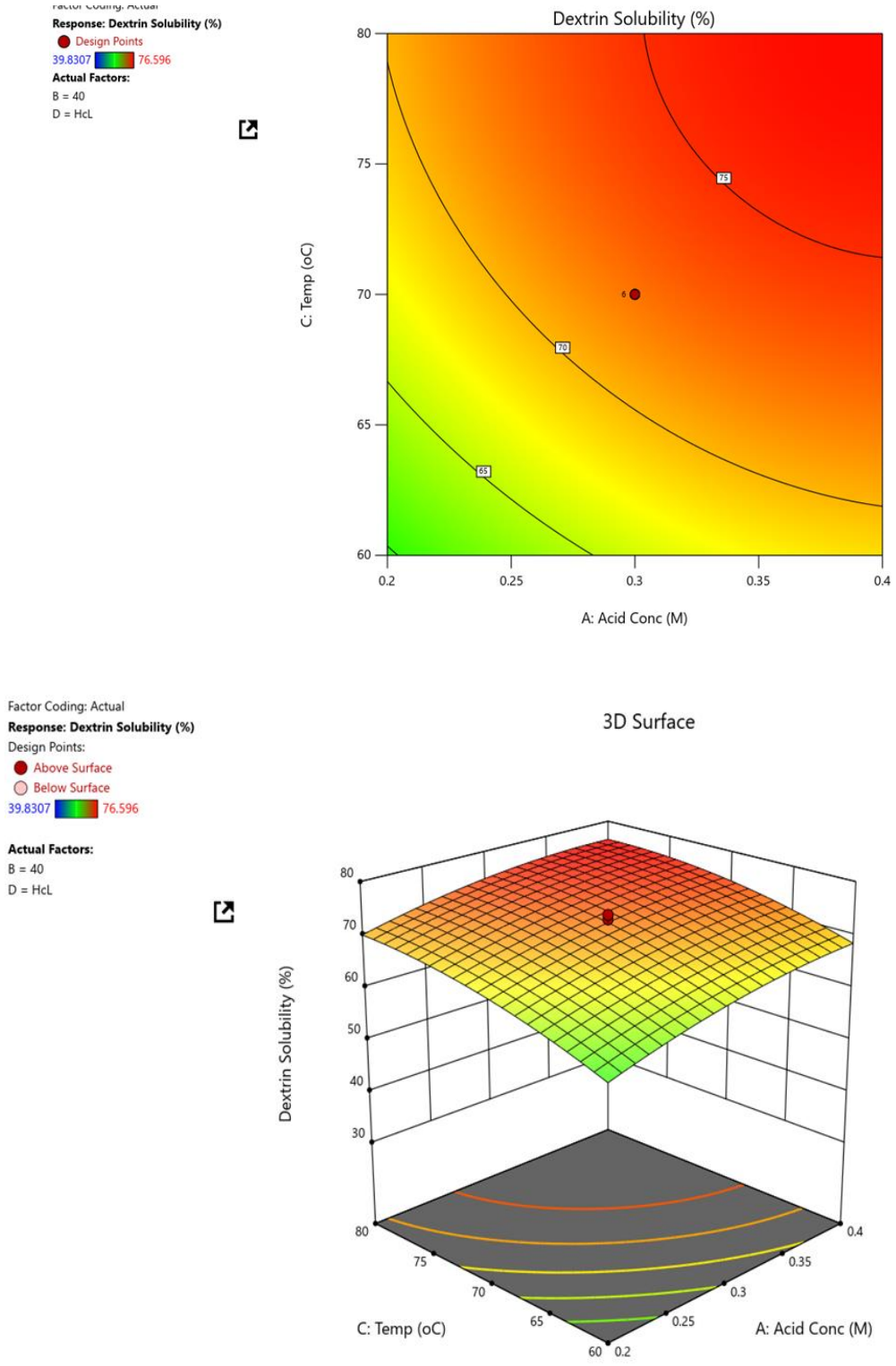

Fig 1: Contour and 3-D interaction plot between Temperature (◦C) and Acid Concentration (M)

Interaction Plot of Time and Acid Concentration: This is the 3-D interaction plot. This plot shows how solubility changes over time at different acid concentrations and the interaction between those factors. At high acid concentration, the solubility rises sharply initially but decreases after a certain time due to over hydrolysis or degradation of dextrin.

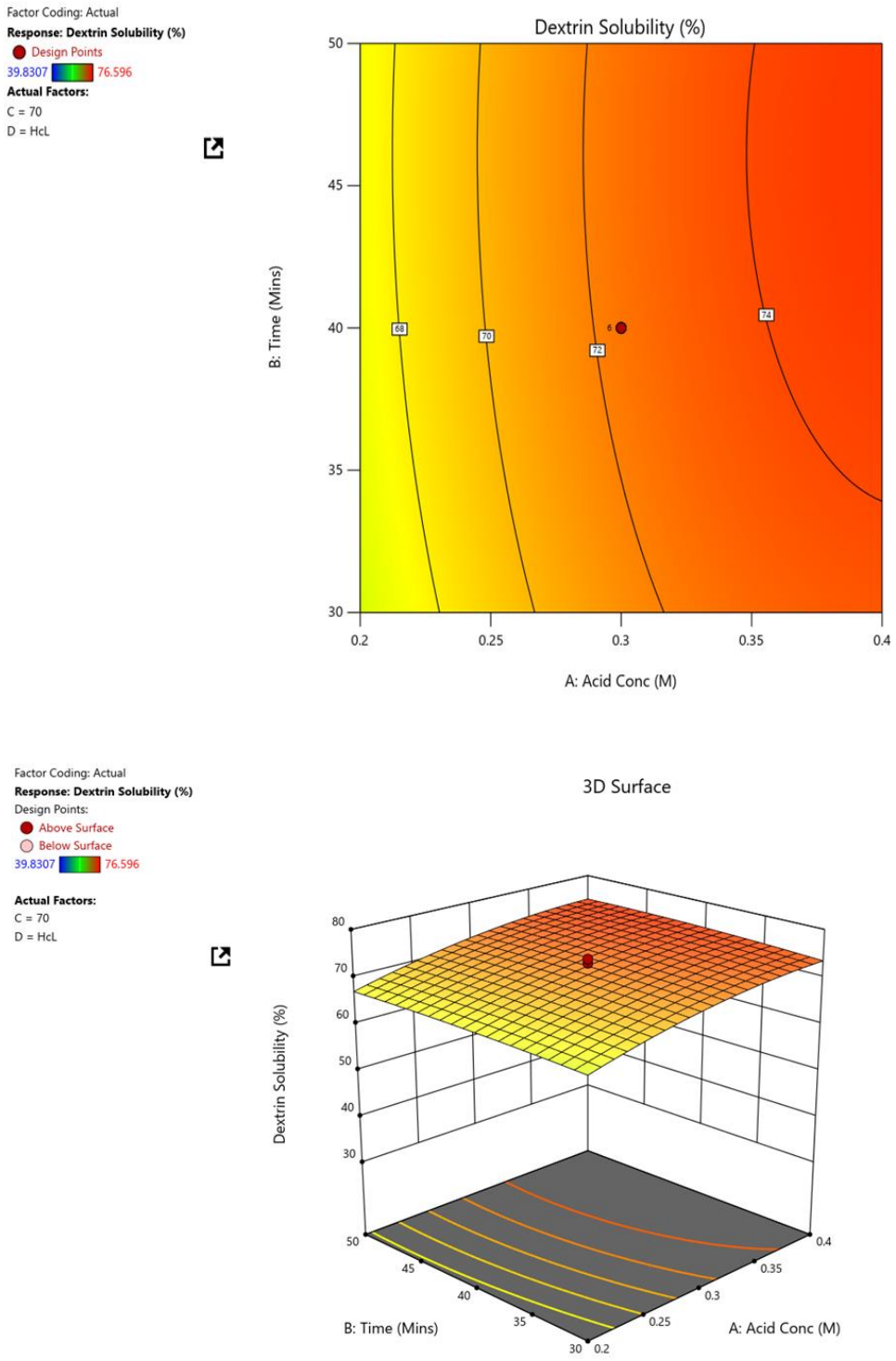

Fig 2: Contour and 3-D interaction plot between Time(Mins) and Acid Concentration (M)

Interaction Plot of Acid Concentration and Temperature: This is the 3-D interaction plot. Understanding the interaction between acid concentration and temperature in relation to dextrin solubility involves balancing these factors to achieve maximum solubility without crossing into conditions where degradation predominates. Initially, as the acid concentration and the temperature were increased the solubility value also increased until a maximum was obtained at the peak of the graph.

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ISSN 2394-7349

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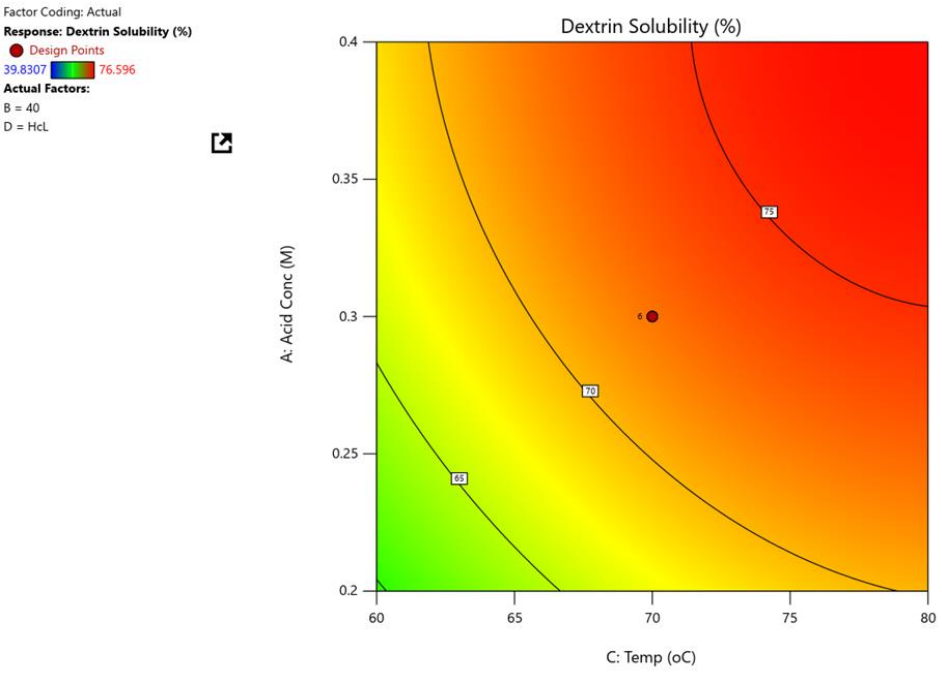

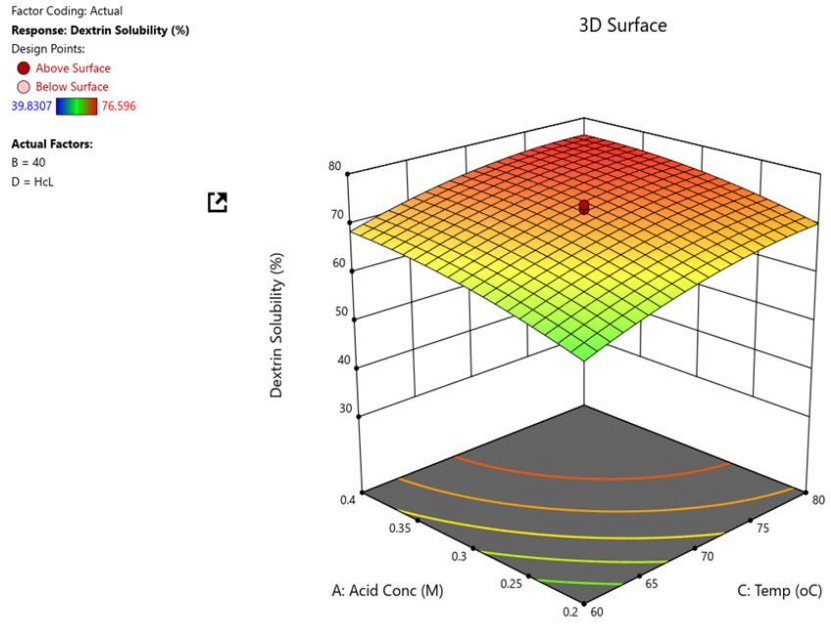

Fig 3: Contour and 3-D interaction plot between Temperature (∘C) and Acid Concentration (M)

TABLE IV: OPTIMAL CONDITIONS

Acid Types	Acid Conc (M)	Time (Mins)	Temp (oC)	Solubility of Dextrin (%)		Error (%)
				Predicted Value	Experimental Value	
HCl	0.392605	46.1343	79.4328	76.465	76.411	0.054
HNO_3	0.365719	46.153	76.1554	53.7225	53.713	0.0095

The result of percentage error showed that the experimental values were closely related to the predicted value. Hence, this research was done properly.

IV. CONCLUSION

Dextrins are produced by hydrolyzing starch through heat, acid, or enzyme treatments. Optimizing the conditions, such as temperature, pH, and reaction time yield dextrins with desired molecular weights and properties. Acid hydrolysis produce dextrins with various levels of depolymerization. Controlling the concentration of acid, temperature, and time helps in producing dextrins with tailored characteristics. Dextrin are used as thickeners, stabilizers, and binding agents. Maltodextrin, a type of dextrin, is commonly used in processed foods. They serve as fillers or carriers in tablet formulations, used in envelope glue, because of their sticky properties when wet, and used as sizing agents in textiles and paper to provide stiffness and improve the finish. Temperature range of 50 to 90°C was adopted while acid concentration range of 0.1 to 0.5 M and time range of 20 to 60 minutes were used for the dextrinization process. The process factors for the dextrinizationn of starch was optimized using Centra Composite Design (CCD) expert Stat-Easy 360.The percentage solubility for nitric acid (HNO_3) gave its highest value at 0.4M concentration, 50minutes and 80^OC was 53.0912%. while HCl gave its highest value at 0.4M concentration, 30minutes and 80^OC was 76.596%. The optimum condition for yellow dextrin was obtained at temperature of 80°C, roasting time of 30 minutes and concentration of 0.4 M and gave a solubility of 76.596% using HCL as acid. From the ANOVA table, P-value less than 0.0500 indicate model terms are significant. In this case A, B, C, D, AC, AD, CD, A^2, B^2, C^2 are significant model terms. The predicted and experimental values of 76.465% and 76.411% for HCl, and 53.7225% and 53.713% for HNO_3 respectively showed good experimental work. From the analysis carried out, temperature, roasting time and concentration has a significant effect on the solubility of dextrin. The adhesive produced was found to be of good quality and can be used for both domestic and building materials applications.

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