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Optimization of Process Parameters for Sodium Carboxymethylcellulose Synthesis from Corncob Using Response Surface Methodolgy

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ABSTRACT
Corncob has proofing record of high reserve in deposit as waste and has high content of cellulose and hemicellulose which can be used as an alternative raw material for synthesis of a valued and widely applied thickener, sodium carboxymethylcellulose (Na-CMC). In this study, Na-CMC was synthesized following the reaction steps which include alkalization and etherification reaction with respect to design of experiment using central composite design of response surface methodology (RSM) to investigate the effect of three variables namely sodium hydroxide concentration, monochloroacetic acid concentration and reaction time on the optimal yield of Na-CMC. The process of carboxmethyl cellulose production was optimized with optimal condition of 23% sodium hydroxide concentration, 78% of monochloroaceticacid concentration and reaction time of 5.0hours. The Na-CMC was characterized by FTIR, XRD and SEM techniques as well as Degree of substitution (DS) was also determined at optimized condition. The synthesized Na-CMC was applied as a gelling agent for ethanol in the formulation of bioethanol gel fuel, as a thickener for water and liquid juice.

Keywords: *Alternative, Biomass, Cellulose, Characterization, Etherification, Thickener, Optimization*

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I. INTRODUCTION

Corncob is regarded as an eco-friendly agricultural waste produced from the harvest of maize plant as a nonedible material separated from the corn grain which has found cheaply available and widely cultivated in different regions in Nigeria most especially in the middle belt. Corncob is highly reached in cellulose fibre containing both cellulose and hemicellulose content of up to 90%[10]. The cellulose extracted from corncob is hydrophilic in nature containing hydroxyl groups in each polymer unit which makes it interactive with metals chemically and open ways for its chemical modification in to sodium carboxymethyl cellulose, methyl cellulose, ethyl cellulose and hydroxypropyl cellulose which are used in medicine, food and pharmaceutical industries [9].

Sodium carboxymethyl cellulose, CMC is one of the most widely applied in food, cosmetic, paint, textile, consumer goods and pharmaceutical [1]. CMC is mainly utilized in food industry as a thickener, stabilizer and binder to improve the texture and stability of various beverages and foods such as ice cream, milk, margarine, candies and peanut butter[4].CMC is a water soluble modified cellulose, biopolymer anionic polysaccharide made by man through the reaction of monochloro acetic acid with alkali cellulose in the presence of ethanol.

Several researches have been conducted on CMC synthesis from cotton, wood pulp, rice straw, banana peel, cassava peel and sea weeds [9]. The optimum condition for CMC synthesis has not been well explained and captured in most of the literature as well as effect and interactions of the factors which form the integral part of this research.

The research aimed at investigating the operating variables or factors determining the optimum condition for optimal yield of Na-CMC using central composite design of response surface methodology.

II. EXPERIMENTAL

Materials

Corncobs were collected from the remained of harvested corn maize after shelling of the cobs in a selected farm at Lafiagi town, Kwara State, Nigeria. The corncobs were first subjected to particle size reduction by first grinding using wooden pestle and a mortar which was finally grounded to particle size of 1.00mm with the help of Lab Miller. The grounded particles were washed to remove sand and other impurities and were later dry inside oven at 60˚C for 3days. Other materials include distilled water, H_2SO_4, methanol, acetic acid, monochloro acetic acid, NaOH, 5.0% Na-hypochlorite and ethanol.

Isolation of cellulose from Corncob

The corn cob pulp was firstly dried to a final moisture content of 10%(w/v) for 3 days in an oven at 60°C. In order to break the fibres, the dried feedstock was grinded to a particle size of ±1.0mm and the pulverized material was soaked in hot water for 1hour and filtered. The residue was treated with 8 % (w/v) NaOH solution at a material to liquid ratio of 1:15 (w/v) at 90°C for three hours in water bath shaker. The cellulose slurry was filtered and washed with distilled water two times. The obtained cellulose fibres was bleached with 5 % sodium hypochlorite at 50°C for 2hours and was subjected to cooling and after, the bleached cellulose fibres was washed with distilled water until it attained a neutral pH, dried in an oven at 60°C for eight hours and weighed using an analytical balance, grounded to powder and kept for further usage.

Figure 1: Cellulose Extraction Stages

Carboxymethyl Cellulose (CMC) Synthesis from Isolated Cellulose

The isolated cellulose from corncob was converted to Na-CMC in two steps, alkalization and etherification of cellulose under heterogeneous conditions. In the alkalization pre-treatment, 10 g of cellulose sample was weighed and added to a 600ml beaker followed by 200 ml of isopropanol. The mixture was left to stand for 5 minutes and 50 ml of aqueous sodium hydroxide concentrations (P) of (20-30%) was added drop-wise while it was stirred in a water bath shaker for an hour. The reaction between the OH of cellulose and NaOH is known as mercerization process. After alkali treatment, etherification reaction was started by the addition of 24mL of monochloro acetic acid (MCA) concentration, Q (70-85%) in the reaction mixture at a temperature of 55°C in a water bath shaker for 3-6 hours of reaction time (R) using Response surface methodology (RSM) obtained from design of experiment (DOE). The slurry was soaked in methanol for 12 hours for ageing or maturation and was later neutralized with 90 % acetic acid where it reached a pH value of 6.5 – 7. The slurry was filtered and then purified by washing with 70% ethanol to remove undesired by product to get pure Na-CMC. Then, the pure Na-CMC was filtered and dried at 60°C in an oven for 24 hours. The total summary of equation of reaction leading to the preparation of Na-CMC and the mechanism of the reaction are given in equations

$[C_6H_7O_2(OH)_3]n + nNaOH \rightarrow [C_6H_7O_2(OH)_2ONa]n + nH_2O$ (1)

Alkali cellulose

$[C_6H_7O_2(OH)_2ONa]n + nClCH_2COONa \rightarrow [C_6H_7O_2(OH)_2OCH_2COONa]n + nNaCl$ (2)

Sodium chloro acetate Sodium caboxymethyl cellulose

$NaOH + ClCHCOONa \rightarrow HOCH_2COONa + NaCl$ (3)

Figure 2: Synthesis of sodium carboxymethyl cellulose from corncob using Water bath Shaker

Determination of Degree of Substitution of Na-CMC

Determination of Degree of Substitution of sodium carboxymethyl cellulose was carried out following the previous method described with some modifications[3]. 5.0g of Na-CMC was mixed with 75ml of 95% ethanol in a beaker and stirred for 5 to 8 minutes. The Na-CMC in the mixture was converted to acidic CMC with addition of 5ml of 2M nitric acid which was heated to boiling for8 minutes and the resulting mixture was subjected to cooling and stirring for 10 to 15mins which led to formation of solid and liquid phase. The solid phase was separated from liquid phase and was washed with 24 ml of 80 % ethanol at 60ºC for 4times and later washed again with anhydrous methanol, filtered and dried at 85˚C for 4 hours and cooled in the desiccator for one hour. The acid CMC obtained was weigh 0.5g and was transferred into 250ml Erlenmeyer flask with 100ml of distilled water added and stirred. The solution was added with 30 ml 0.3 M NaOH which was boil for 20 minutes and the dissolved mixture was titrated with 0.3 M HCl where the colour change from dark pink to colorless was observed after Phenolphthalein indicator has been added. Equation (3) and (4) give summary for calculation of DS.

$$A = \frac{MN-PO}{R} \quad (4)$$

$$DS = \frac{0.162\,X\,A}{1-(0.058\,X\,A)} \quad (5)$$

Where,

A = milli-equivalents of consumed acid per gram of specimen
M = volume of Sodium hydroxide added

N = concentration in normality of sodium hydroxide added

P = volume of consumed chloric acid

Q = concentration in normality of chloric acid used

R = specimen grams used
Molecular weight of the anhydrous glucose unit = 162
Molecular weight of carboxymethyl group.=58

Behaviour of Ethanol and Liquid Foods thickened by Na-CMC and their viscosities

The behavior of ethanol and liquid food samples comprised of water, milk and orange juice were influenced by the effect of Na-CMC on them. These was achieved by addition of 2% (w/v) of Na-CMC, slowly pouring on the liquid samples and were stirred at 300rpm for 10mins per each. The viscosities of the samples were finally measured using Thermo scientific viscometer equipped with spindle R27 and rotation speed of 55rpm.

Fourier transformed infrared spectroscopy (FTIR)

The Na-CMC produced from the extracted cellulose was subjected to test to find out and verify its functional group with the use of Fourier Transform Infrared Spectrophotometer (FTIR**).**The Na-CMC sample was first dried in the oven at 60 ºC for moisture removal and after 2.0mg of the sample together with 0.2mg of potassium bromide,

KBr were mixed, finely grounded and compressed to form a transparent pellet. The samples were dried in an oven at 60 ºC to remove the moisture. About 0.2 mg of sample and 2 mg of potassium bromide were mixed and ground finely before the mixture was compressed to form a transparent pellet. The infrared spectra of these samples were measured in the transmission of a wavelength number range between 4000 and 400 cm^{-1}.

Scanning Electron Microscope

The surface morphologies of Na-CMC was examined by SEM and Sample was coated with a thin layer of conducting material (gold) and imaged at ×7000 magnifications with 15 kV accelerating voltage at a pressure of 70 Pa. A focused high energy beam of electron was interacted with the surface of sample and generated a secondary electron, back scattered electron with characteristic X-rays signals were indicated. These signals were perceived by the detector and images were displayed on the cathode ray tube screen.

X-Ray Diffraction (X-RD)

Degree of crystallinity of the Na-CMC was determined using X-ray Diffraction. X-Ray diffraction patterns of the Na-CMC sample was achieved through X-Ray diffractometer equipped with CuKα radiation in the 2θ region with a scan step time of 37s and 0.026°. Sample was prepared by pressing the powders between two glass slides into a flattened sheet. The X-Ray patterns were taken by using radiation source CuK by supplying 40 kV and 40mA to X-ray generator. The patterns were recorded at 2θ from 10° to 90°.

III. RESULTS AND DISCUSSION

Table 1: Independent parameters with corresponding coded levels for Gelling Agents Production from Corncob (Na-CMC) using Central Composite Design

Factor	Notation	Units	Level				
			-α	-1	0	+1	+α
Sodium Hydroxide concentration	P	%	18.30	20.0	22.5	25.0	26.70
Chloroacetic acid concentration	Q	%	64.89	70.0	77.5	85.0	90.11
Reaction Time	R	hr	1.98	3.0	4.5	6.0	7.02

Table 2: Result of Optimization of Gelling Agent Production from corn cob using Central Composite Design

Run Order	Pt. Type	Blocks	P	Q	R	Viscosity S(kmpa-s)	DS
1	0	1	23	78	5	4875	0.82
2	-1	1	23	78	1.98	1940	0.20
3	0	1	23	78	5	4875	0.82
4	0	1	23	78	5	4875	0.82
5	-1	1	26.70	78	5	4480	0.60
6	0	1	23	78	5	4875	0.82
7	1	1	25	85	3	3295	0.40
8	0	1	23	78	5	4875	0.82
9	1	1	20	70	3	2150	0.30
10	-1	1	23	64.89	5	2075	0.27
11	1	1	20	85	6	4055	0.55
12	-1	1	18.30	78	5	3600	0.45
13	1	1	25	70	6	3045	0.33
14	-1	1	23	90.11	5	3900	0.49
15	1	1	20	85	3	3100	0.35
16	1	1	20	70	6	3100	0.35
17	1	1	25	70	3	2000	0.21
18	-1	1	23	78	7.02	4085	0.59
19	0	1	23	78	5	4875	0.82
20	1	1	25	85	6	4560	0.74

DS: Degree of substitution

Table 3: Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	221385707	2459834	174.2	0.000
Linear	3	8625907	2875302	203.77	0.000
P	1	229096	229096	16.24	0.002
Q	1	4089063	4089063	289.79	0.000
R	1	4361753	4361753	309.11	0.000

Square	3	9824863	3274954	232.09	0.000
P^2	1	1069338	1069338	75.78	0.000
Q^2	1	6072910	6072910	430.38	0.000
R^2	1	4702601	4702601	333.27	0.000
2-Way Interaction	3	163847	54616	3.87	0.045
PQ	1	101597	101597	7.20	0.023
PR	1	55807	55807	3.95	0.075
QR	1	3732	3732	0.26	0.618
Error	10	141107	14111		
Lack-of-Fit	5	141107	28221		0.724
Pure Error	5	0	0		
Total	19	22279614			

S	R-sq	R-sq(adj)	R-sq(pred)
118.788	99.37%	98.80%	94.87%

The model equation's significance was checked by statistical analysis of Variance (ANOVA). The value of P from the model shows that regression model is significant (p =0.000) and that is, at least one of the terms in the model has an impact on the gelling agent yield. ANOVA results presented in the Table 3 showed that all the three linear terms, P, Q and R are very significant on the respond yield, S (gelling agent).The squared or quadratic term P^2, Q^2 and R^2 as well as the interactive term PQ were very significant and whereas the PR and QR were not significant on the response S (gelling agent yield). For gelling agent production from corncob, sodium carboxymethyl cellulose (Na-CMC), the estimation of adjusted square correlation coefficient, R-Sq(adj) of 98.80% was additionally higher, which demonstrated the higher essentialness of the model. The R-Sq (pred) estimation of 94.87% demonstrated the sensible concurrence with the "Adj R-Sq" esteem of 98.80%. This demonstrated a very good decent assent between the watched and the anticipated qualities [11].

Table 4:Regression Coefficient

Term	Effect	Coef	SE Coef	T-Value	P-Value	VIF
Constant		4698.0	53.0	88.70	0.000	
P	255.90	127.90	31.70	4.03	0.002	1.05
Q	1120.20	560.10	32.90	17.02	0.000	1.04
R	1134.10	567.0	32.30	17.58	0.000	1.04
P^2	-499.20	-249.60	28.70	-8.71	0.000	1.05
Q^2	-1329.50	-664.80	32.0	-20.75	0.000	1.03
R^2	-1247.50	-623.80	34.20	-18.26	0.000	1.06
PQ	223.40	111.70	41.60	2.68	0.023	1.01
PR	161.80	80.90	40.70	1.99	0.075	1.05
QR	42.20	21.10	41.10	0.51	0.618	1.03

From the regression coefficient in the table 4 above, the linear terms P, Q and R showed very significant on the gelling agent yield (S). The squared or quadratic terms M^2, N^2 and Q^2 were very significant on the yield of the gelling agent (Na-CMC). The interactive term PQ is very significant and whereas PR and QR were not significant on the respond yield(S). The values obtained showed some differences in the significant of process parameters compare to the previous findings [9]. The above implies that the model equation for Optimization of gelling agent production from corncob can be written as;

$S = -87526 + 1289\ P + 1764\ Q + 2242\ R - 39.93\ P^2 - 11.818\ Q^2 - 277.2\ R^2 + 5.96\ P*Q + 21.6\ P*R + 1.88\ Q*R$

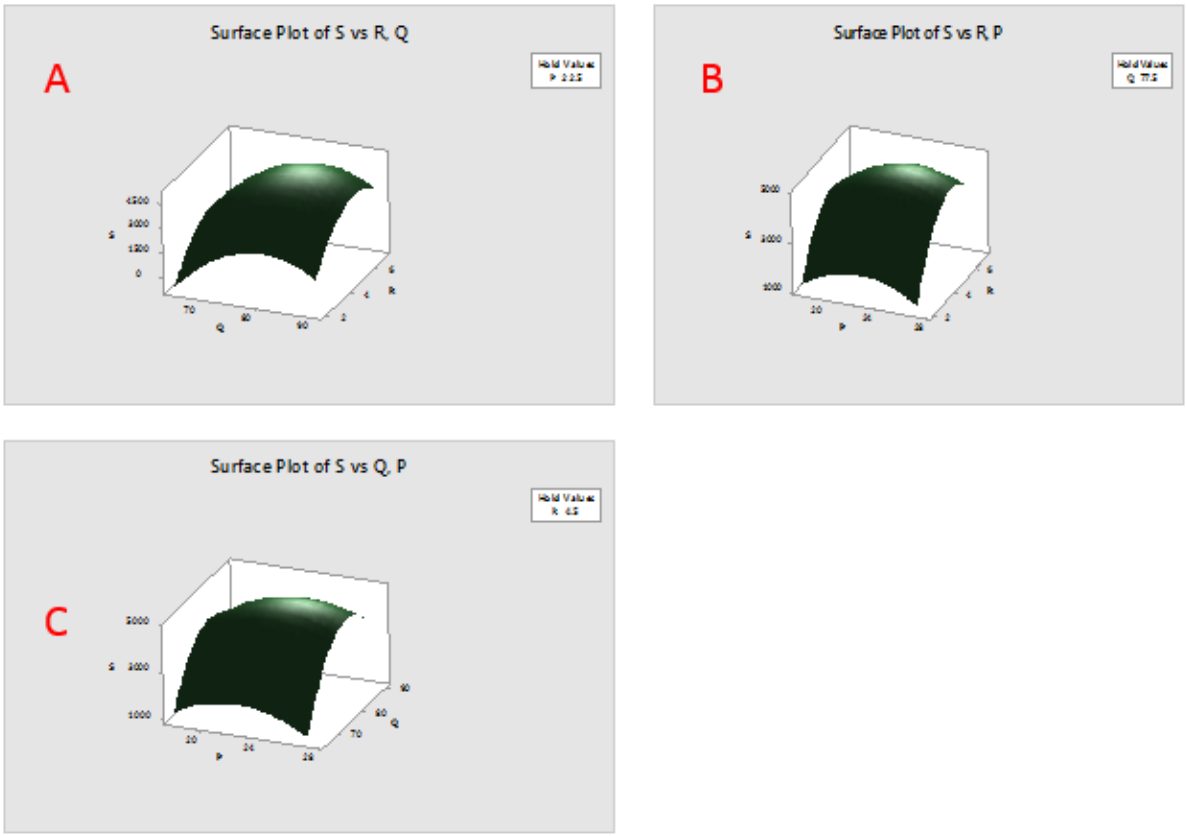

Figure3:The 3D Surface plot showing (A): the effect of monochloro acetic acid concentration (Q) and reaction time (R)on the gelling agent yield(S); (B):the effect of sodium hydroxide concentration(P) and reaction time (R) on the gelling agent yield(S);(C)::the effect of sodium hydroxide concentration(P) and monochloro acetic acid concentration(Q) on the gelling agent yield(S)

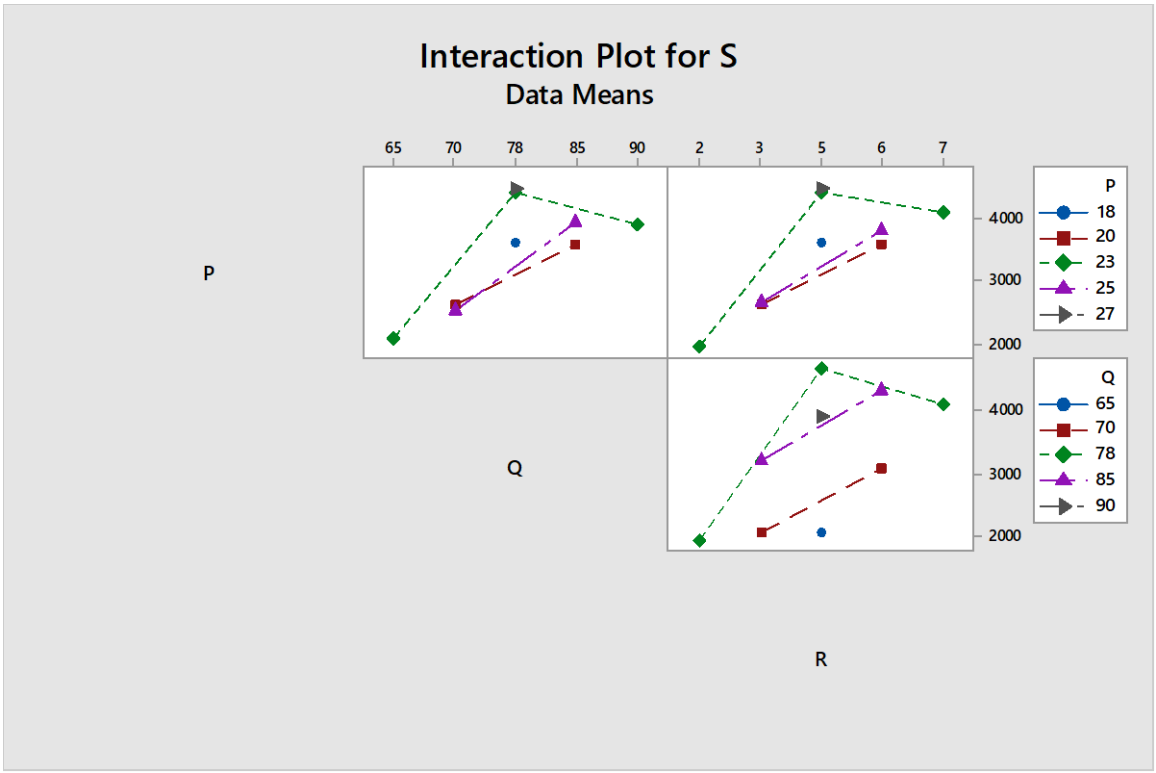

Figure 4: The Interaction Plot of Gelling Agent Yield from Corncob(Na-CMC)

Interaction Plot

Figure 4 indicated row 1 matrix plot which comprised of sodium hydroxide concentration (P) and chloro acetic acid concentration (Q) showed good significant interaction. The gelling agent yield increases maximally at a level of P with green line which contain a value of 23% corresponding to the level of Q at 78%. The gelling agent yield is followed by the concentration of P with a purple line which contained a level of 25% with a corresponding Q value of 85% and also the least respond yield at a p value with a pink line of 20% which corresponded to 85% value of Q . The Row 1 matrix of P and R which comprised of sodium hydroxide concentration (P) and reaction time (R) showed fairly significant interaction at a factor of P with a green line at a level of 23% and at R with the corresponding level of 5hours. The remained two levels of R (Purple and pink lines) indicated little interaction signifying no significant yield of the gelling agent. The matrix plot of Q and R showed less interaction at the Q level of 78% which corresponded to the value of R at 5.ohours. The remained two lines of Q are almost parallel and exhibit no interaction because the levels does not effect changes by the level of Q and led to constant value of response in gelling agent yield.

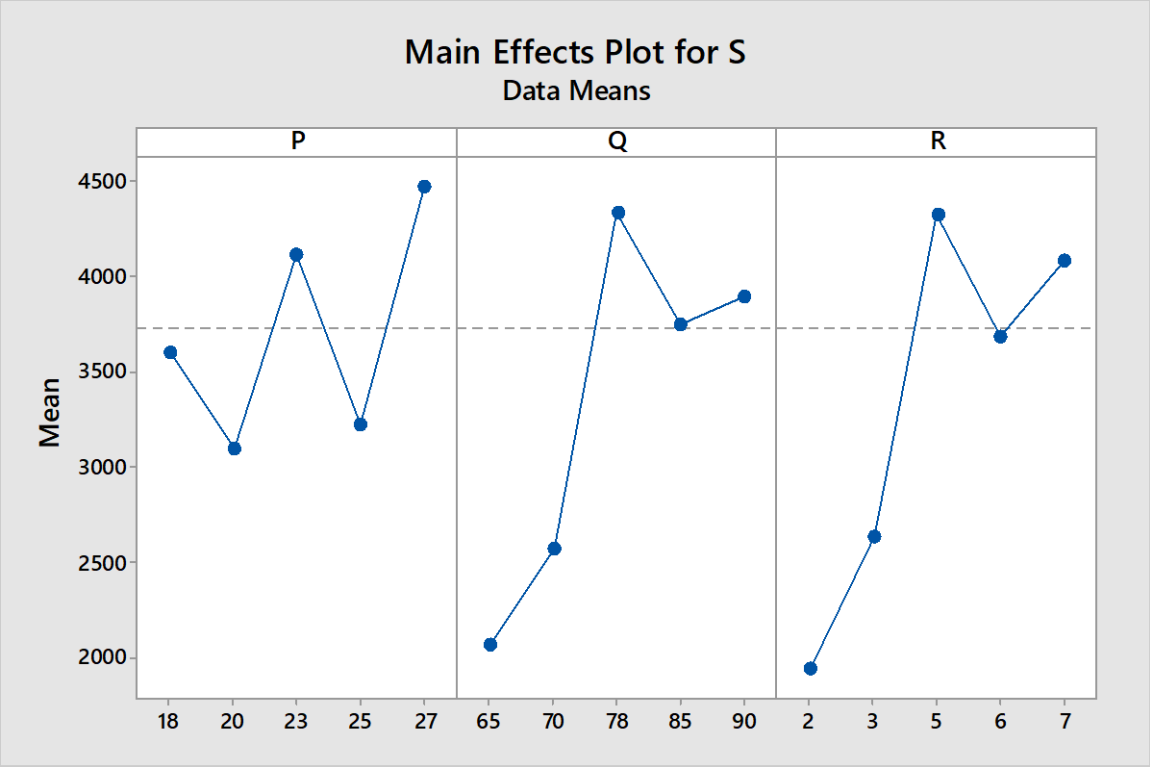

Figure 5: Main Effects Plot of Gelling Agent Yield from Corncob(Na-CMC)

Main Effects Plot

The main effects plot is use to look at contrasts between level means for one or more elements. There is a main effect when diverse levels of an element influence the response in a different way. A main effect plots diagrams of the response for every component level associated by a line. Figure 5 showed there is gradual increase in mean response with additional increases in sodium hydroxide concentration(P) time from 20 to 23%. It however showed that further increase in P value to 25% reduces the mean response of the gelling agent yield. The chloro acetic acid concentration (Q) played a great role in high yield of the gelling agent. There is gradual increase in the mean response of gelling agent for the value of Q from 65 to70%. The gelling agent yield increases further and attained optimal point with additional increases in the value of Q to 78% and where subsequent increment in Q drops down the gelling agent yield. This shows that the mean response on gelling agent yield decreases with further increases in chloro acetic acid concentration to a value somewhere close to 85% and above. There is effect in response in gelling agent yield with reaction time(R) of the medium. The is gradual increase in the gelling agent yield at a value of reaction time from 2.0 to 3.0 hours and increases additionally to an optimum point at R value of 5.0 hours. Further increases in R reduces the response in gelling agent yield to an R value of 6.0. This plot demonstrates a reasonable diverse in size of the impact on the response among the elements: P, Q and R. The variable R had the best impact on Y, trailed by Q and finally P. The positive estimations of the impact demonstrate that the expansion in the extent of parameter builds the response Y.

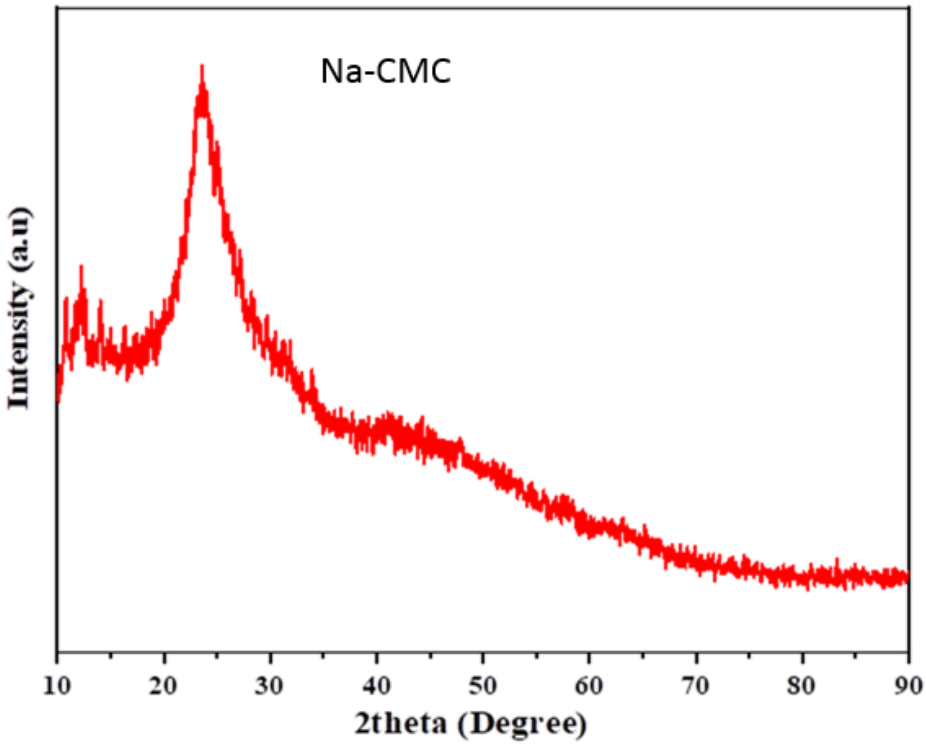

Figure 6: XRD Pattern of Na-CMC

The Figure 6 shown above indicated the XRD pattern of the Na-CMC microstructure. The diffratogram of the synthesized CMC displayed main peaks at 2θ =13.5˚, 22.5˚, 25.0˚ and27.5˚respectivelycorrespond to the position of crystallographic planes indicating transformation of cellulose during alkalization process which has led to formation of Na-CMC[13].The replacement of high sharp peaks with wide broad spectrum peak are indications of complete modification of alkali cellulose to Na-CMC and hence characteristic diffraction pattern of crystalline peaks of CMC [6].This means that carboxymethyl cellulose crystallinity has been partially transformed into amorphous phase. This is due to the substitution of the OH groups in cellulose with CH3COO– groups [7].

FT-IR Analysis of Corncob Gelling Agent(Na-CMC)

The FTIR spectrum of CMC shows the presence of carboxyl (COO-) group at 1558-1651 cm^{-1} and 1416-1449cm^{-1}. carboxyl groups and its salts show two peaks at wavenumber 1600-1651cm^{-1} and 1415-1449cm^{-1} which indicates the presence of carboxymethyl substituent (COO^-) and could be used as an evidence to indicate the replacement of hydroxyl groups with carboxyl group when the carboxymethylation reaction occurred[8].The bands at 1416cm^{-1} and 1326cm^{-1}in Figure 7 are attributed to CH_2 scissoring and OH bending vibrations respectively. The C-O-C glycosidic ether band at 1084-1043cm^{-1}arising from the polysaccharide components [5]. In addition, the peak at 879 cm is associated with the β-(1,4)-glycosidic linkages between the glucose units in cellulose [3]. The result of the FTIR obtained was in agreement to that reported previously [13,;8&7]

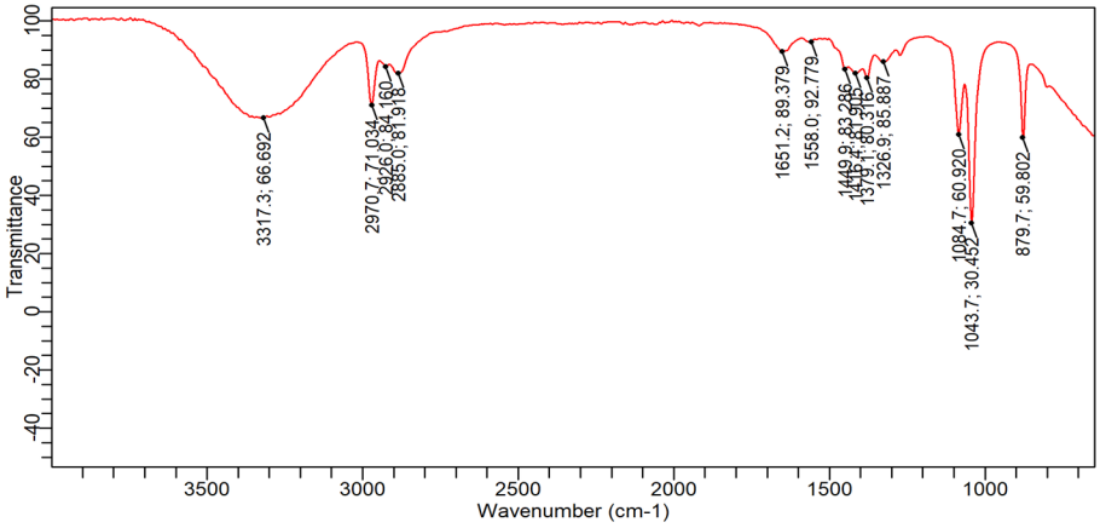

Figure 7: FT-IR of Gelling Agent Yield from Corncob(Na-CMC)

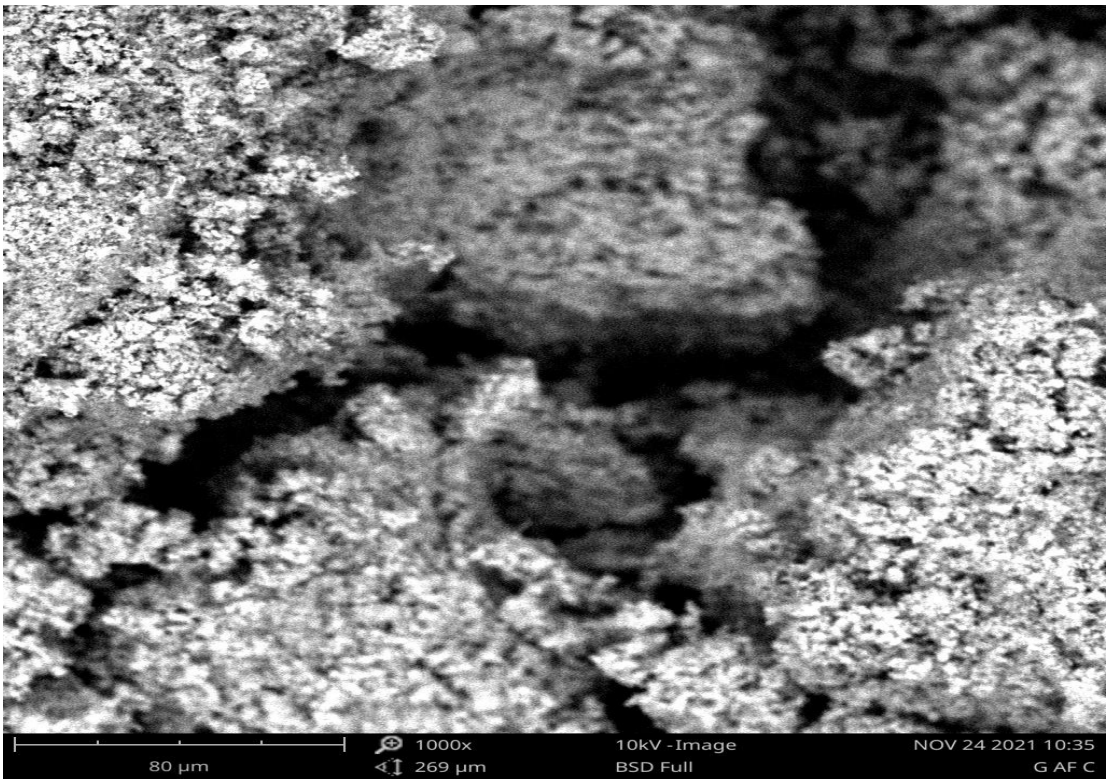

Figure 8: SEM of Gelling Agent from Corncob (Na-CMC)

Figure 8 shows SEM micrograph of the Gelling agent from Corncob(Na-CMC) at higher magnifications and revealed that CMC had a rough surface with a partial round fibrils and not elongated like cellulose structures, resembling particle of atoms closely packed together to form cluster [7]. This morphological characteristic is associated with large amount of carboxymethyl group initiated in to cellulose fibre surface during etherification process of Na-CMC formation[12].

Table5: Behaviour of Ethanol and Liquid Foods thickened by Na-CMC and their viscosities

Sample	Initial Viscosity(cp)	Final viscosity(cp) + 2% Na-CMC	Increased in viscosity	Initial Behaviour	Final Behaviour
Ethanol	0.947	750.53	749.583	Thin liquid	Honey like
Water	0.798	204.20	203.40	Thin liquid	Nectar
Orange Juice	2.52	495.05	492.53	Thin liquid	Honey like
Milk	1.975	401.85	399.87	Thin liquid	Honey like

The table 5 above showed the behavioural pattern of ethanol and liquid foods thickened by Na-CMC after the experiment conducted, it was observed that there is viscosity increase in the final products after been thickened and behavioural changes in the products were noted and revealed that the viscosities of the products were enhanced and modified from thin liquids to nectar and honey like structure[2]. Based on these behavioural changes, Na-CMC can be applied as a thickener not only for ethanol but in food formulation as a thickener for water, milk and orange juices.

IV. Conclusion

Optimization of process parameters for synthesis of sodium carboxymethyl cellulose was successful carried out. The optimum conditions for optimal yield of Na-CMC was realized at the viscosity value of 4875mPa-s and degree of substitution of 0.82 which corresponded to 23.0% of sodium hydroxide concentration (P), 78.0% of monochloro acetic acid concentration(Q) and 5.0hr of reaction time (R) respectively. The ANOVA results presented showed that all the three linear terms, P, Q and R are very significant on the respond yield, S (gelling agent).The squared or quadratic term P^2, Q^2 and R^2 as well as the interactive term PQ were very significant and whereas the PR and QR were not significant on the response S (gelling agent yield). For gelling agent production from corncob, sodium carboxymethyl cellulose (Na-CMC), the estimation of adjusted square correlation coefficient, R-Sq(adj) of 98.80% was additionally higher, which demonstrated the higher essentialness of the model. The R-Sq (pred) estimation of 94.87% demonstrated the sensible concurrence with the "Adj R-Sq" esteem of 98.80%.The behavioural changes were observed on ethanol and liquid foods thickened by Na-CMC and has confirmed it as a good thickener.

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