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Design Process of CSTR for Production Carboxyl Methyl Cellulose

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***Abstract* - The aim of this study is to generate tools for utilizing Cellulose Biomasses Polymers from Palm Frond Fabricating Unit Design, Principles of material balance and volume equations were perform on the Reactor system to derive model equations applied in obtaining the reactor design parameters. Synthesize and characterize of carboxyl methyl cellulose (CMC) from Phoenix dactyliferaL, Date palm (leaves by etherification reaction using sodium mono chloroacetic acid (MCA)) and sodium hydroxide. Crystallinity of purified cellulose and (CMC) with degree of substitution of 0.77 and a percentage of produced CMC was 71%. The hydrolysis and dehydration reaction of Cellulose and Sodium hydroxide and Sodium monochord acetic acid respectively to Caboxylmethylcellulose is Exothermic. A cooling jacket incorporated to account for the supply and treatment of heat liberated. As a result, an integrated design of the CSTR reactor was presented to produce 150 tons/year of CMC with a volume of 2435.26 dcm^3.**

Keywords: Caboxylmethylcellulose, Cellulose, Date Palm, Isothermal, CSTR design, Material Equation.

I. INTRODUCTION

Design is a creative activity that integrates the elements of art and science to create something new or retrofit for existence. This research focuses on the industrial fabricating and industry unit design of Isolated Polymers from Local materials that is applicable for industrial applications in order to increase the national income by adding a natural resource and to decrease the cost and dependency on imported polymers from abroad. For these purposes and during the last century and a half, new families of engineering materials (known as polymers) have discovered and produced. Polymers are at the basis of important industrial goods. Their rapid growth in production caused (beside social factors) by the necessity to replace classical materials [1].

In nature, Cellulose is the most abundant polymer on earth, which makes it most common organic compound. Annual cellulose synthesis by plants is close to1012tons. Plant contains approximately 33% cellulose, whereas wood contains 40-50% and cotton80- 95%, Newspaper 40-55%palm frond 46-48%.Most of cellulose utilized as a raw material in paper production this equator annually. From this, only 4 million tons used for further chemical processing annually [2]. Carboxyl methyl Cellulose (CMC) is one of the most promising cellulose derivatives. Many research articles reported on CMC, depending on their sources and application fields. CMC obtained by chemical modification of natural cellulose which considered a linear long chain water-soluble ionic polysaccharide as in figure (1)[3].

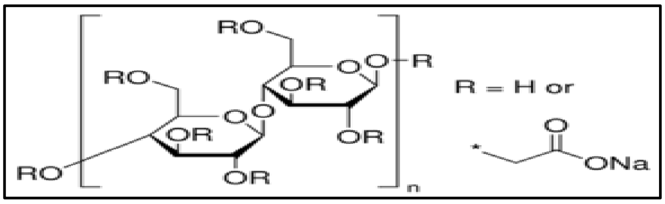

Figure 1: Molecular structure Carboxymethylcellulose sodium salt cellulose derivative that is a beta-(1, 4)-D-glucopyranose polymer. IUPAC name: Sodium 2 choloroacetate

In the chemical field, many methods for the preparation of CMC have described but mainly they divided into two groups. Solid-phase obtained in industry without solvents or in suspension phase, the technological process for obtaining carboxyl methyl cellulose consists by reacting the free hydroxyl group in the AnhydrousGlucose Units (AGU) with various chemical substitution groups. The introduction of substituent disturbance the intermolecular and intermolecular hydrogen bonds in cellulose, which leads to liberation of hydrophilic character of numerous hydroxyl groups and restriction of the chains to close associate.CMC usually prepare from agricultural wastes like Rice straw, corn husk, Cotton, wheat straw, Sugarcane, Baggas, Mesquite, Date palm, etc. The following stages for technological process are: Treatment of cellulose with alkali, Processing with powder of monochloroacetic acid (MCA) or (Na-MCA), Carboxylation of cellulose, Drying and finally Grinding [4][5].CMC has received the greatest of all water-soluble cellulose ethers of

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practical importance; cellulose production organized in most industrialized countries. The largest and most widely used is CMC, which has a degree of substitution of 0.6-0.9.[6]. The CMC can be neutralized and dried immediately to give a technical grade or neutralized and washes to give a purified grade CMC that has many interesting properties when dissolved in aqueous solution, this will depend on the CMC grade and the solution condition [7].

The synthesis process to produce CMC takes place in CSTR reactors reaction. Therefore, the kinetics of CMC synthesis to determine the rate constants of synthesis reactions must know how to simulate, model and develop of a mathematical CSTR reactor. The object of the study is to fabricate the CMC synthesis reactor, and the purpose of this work is to develop a mathematical model of synthesis reactor based on the model of the kinetics of CMC production.

Synthesis of Carboxymethylcellulose (CMC) made in a Continuous Stirred Tank Reactor (CSTR) design. Chemical kinetics, Thermodynamics, fluid mechanics, mass transfer, heat transfer as well as economics must complement these. The perfect mixing attained by the effect of a stirrer in the reactor for that purpose ensures contents of the reactor properly mixed, that mean the temperature and concentration of the content are the same at the product in the reactor.

Application of CMC

CMC widely used in various industries like oil and gas, textile, mining and processing, construction, perfumed and cosmetic, food and others. The use of CMC in these areas is highly dependent on purity, degree of Substitution, degree of polymerization and homogeneity, which determine the characteristic. As CMC is non-toxic component, typically, CMC products classified into three different grades: industrial grade, pharmaceutical grade and food grade based on purity and degree of substitution. Despite the large variety of cellulose derivatives that have made, there is continuous expansion on the worldwide market of cellulose ethers because of their economic efficiency, availability and easy handing.

Synthesized materials are carboxymethylcellulose (CMC) with a wide range of uses in paper industry, in food products [8], but its water-soluble derivatives have found various applications in food, pharmaceutical, detergents and increased the accessibility of fibers to chemicals by cosmetics industries. The effect of CMC addition on the rheological properties of betonies clay suspensions has been studied and experimented thoroughly [9]. In the same manner, as a preservative for swelling. Furthermore, in recent years, polymer composites and Nano composites have attracted great attention as excellent materials for removal of heavy metals as well as for treatment of wastes.[10].

Likewise, CMC used as drilling fluid system at Block 6 Balila field in South West Sudan and found that the drilling fluid rheological properties improved. There was an increase in fluid viscosity and the filtration volume decreased and a good fluid performance in improving drilling efficiency [11].

II. MATERIALS AND METHOD

Sample Collection and Pretreatments

The Palm Frond fibers raw materials or Raw-dry date palm leaves (Phoenix dactylifera L.), collected, washed, air-dried and ground using an electrical grinder.

Purification of Cellulose from Date Palm (Phoenix dactylifera L.) leaves

100 grams of the leaves were soaked in a 500ml solution of 1M (4% NaOH), at 80Cº for 4 hours with constant stirring. Sodium hydroxide solution removed by decantation and the content washed several times with distilled water. This step repeated four times until the color of the supernatant turned pale yellow. The resulting product bleached using a mixture of Clorox solution (I liter of 1.7% NaOCl) with a buffer solution (1 liter of a mixture of 27 grams of sodium hydroxide and 75 ml of concentrated acetic acid) at 80 Cº for 4 hours. The bleaching step repeated four times finally the product washed thoroughly with distilled water. The product left to dry first at room temperature and then at 100 Cºusing oven.

Optimization of the Carboxyl methylation of Cellulose

2 grams of purified cellulose suspended in 40 ml of Iso-propanol and stirred continuously. 10 mL of 20% (w/v) NaOH solution were added drop wise over a period of 30 min. The mixture stirred for one hour at room temperature. 6 grams of monochord acetic acid (MCAA) dissolved in 10 mL of isopropanol has added to the alkalized cellulose and the temperature was raised to 55 Cº. The reaction allowed to continue for 2 hours then neutralized with 90% acetic acid and filtered. The obtained CMC was washed with 70% ethanol five times to remove undesirable materials and dried at 60 Cº in an oven the above experiment was repeated using the same 20% concentration of sodium hydroxide but varying reaction times of 3 and 4 hours[12].

Synthesis of Sodium Carboxyl Methyl Cellulose at Optimum Conditions

Absolute values of degree of substitution (DS) of all samples obtained and recorded; the sample with the highest DS has chosen and synthesized for further analysis. For

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fabrication of CMC at optimum conditions reaction, exactly typical conditions has followed, figure 2. The condition is 2gm of cellulose, 6 grams of MCAA, 40 % NaoH, Temperature 55 C°. X-Ray diffraction (XRD) patterns obtained to investigate the variation in crystallinity of purified cellulose and carboxymethyl cellulose (CMC) with DS of 0.77, the percentage of produced CMC was 71 %.

Figure 2: Process of Local CMC Extraction

Kinetics, Rate Law and Process Chemistry

Synthesis of Carboxymethylcellulose (CMC)

In practice, the manufacturing of CMC involves two steps, the first is treating the cellulose with NaOH, and the alkali cellulose is accessible and reactive toward MCA that added to reaction as a second step [13]. The reaction between alkali cellulose and the etherification agent carried out in aqueous system. Converting all this laboratory application into chemical equations according to the following reactions:

Cellulose + Sodium Hydraulic → Hydrolysis Cellulose

$C_6H_7O_2(OH)_3+NaOH \leftrightarrow C_6H_7O_2(OH)_2ONa+H_2O$ (Reaction 1)

Hydrolysis Cellulose + sodium mono chloride acid → Carboxyl methyl Cellulose + chloride Sodium + water

$C_6H_7O_2(OH)_2ONa+ClCH_2COONa \rightarrow C_6H_7O_2(OH)_2OCH_2COONa+NaCl+H_2O$ (Reaction 2)

$ClCH_2COONa+NaOH \rightarrow HOCH_2COONa+NaCl$ (Reaction 3)

One of the main advantages of the solid-phase method for producing CMC is the absence of production waste and fire safety. Although, the disadvantage of the solid-phase method is the complexity of predicting the parameters of the final production.

The synthesis process takes place in CSTR reactor equipped with screw devices that serve to move the reaction mass. Therefore, to determine the synthesis reaction constants that are essential for the mathematical modeling calculations required to design the required reactor.

Kinetic scheme for Carboxyl Methyl Cellulose

For all technological processes in order to obtain carboxymethyl cellulose, the chemical core is alkaline cellulose (reaction (1)), the reaction of alkaline cellulose with monochloroacetic acid or Sodium Monochloroacetate (reaction (2)) to achieve CMC. The major reaction of the formation of CMC, NaCl, H_2O Throughout the Carboxymethylation of cellulose and a side reaction of the hydrolysis of SMCA with the formation of sodium glycolate (HNA) and NaCl reaction (3) as and the following reaction

$C_6H_7O_2(OH)_3+NaOH \leftrightarrow C_6H_7O_2(OH)_2ONa+H_2O$,

$C_6H_7O_2(OH)_2ONa+ClCH2COONa \rightarrow C_6H_7O_2(OH)_2OCH_2COONa+NaCl$,

$ClCH2COONa+NaOH \rightarrow HOCH2COONa+NaCl$

A formal form of kinetic diagram:

$R\text{-}OH + NaOH \leftrightarrow R\text{-}ONa+H_2O$ (Reaction 4)

$R\text{-}ONa+MAH \rightarrow CMC+NaCl$ (Reaction 5)

$SMCA+NaOH \rightarrow HNa+NaCl$ (Reaction 6)

The kinetics of the process known to be the fundamental for modeling a chemical reactor, as a consequence, the accuracy of all subsequent calculations depends on the accuracy with which the kinetic equations describe the process. The kinetics of CMC reactions when studied are as follows:

Cellulose → Hydrolysis Cellulose → Carboxyl Methyl Cellulose

Let: Cellulose = A, Hydrolysis Cellulose = B, Carboxyl Methyl Cellulose= C

Rate of Cellulose = rA = $dC_A/dt = KC_A$,CA= Final concentration of A

Rate of Cellulose Hydrolysis = rB = $dC_B/dt = KC_B$,C_B= Final concentration of B

Rate of CarboxyL methyl Cellulose= $r_C = dC_C/dt = KC_C$, C_C= Final concentration of C[14].

To measure the values of the kinetic constants Ki, corresponding to the value of described by the equation of Arrhenius, determined activation energy (E) is equal to 3,088.10 4 J/mol, pre-exponential factor (A) is equal to

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1,641.10 3 1/sec and specific thermal effect (QP) is equal to 1789 kJ/kg for etherification of CMC[15].

III. METHODOLOGY

Development of reactor Model

From material balance equation, that applies the principle of mass conservation where the starting point of reactor design the principle states that:

Rate of accumulation of reactant within element of volume

d (Ci V) /dt

Rate of reactants loss due chemical reaction = $(-r_{i)}\ V_R$

Rate of reactants flow out of element of volume = $F_{i.o}\ (1-X_i)$

Substitute all previous reactions (reaction 1 to reaction 6) into the substance flow rate law as:

$F_{i.o}$ =Initial molar flow rate of specie (i) = $F_{i.o}(1-X_i) + (-r_i)$ VR + d/dt (C_iV)

$F_{A.o}$ =Initial molar flow rate of specie= $F_{A.o}(1-X_A) + (-r_A)\ V_R$ + d/dt (C_AV)

X_i = Fractional conversion of specie i.

Assumptions for the reactor

- Steady state operation
- The feed is properly mixed or well stirred.
- Isothermal and adiabatic system.
- Constant density system.
- Cylindrical tank with hemispherical head.

Applying the assumptions where necessary gives:

$F_{A.O}= FA_{.O}\ (1-X_A) + -rA\ V_R$

$V_R = F_{A.O}X_{A\,/}\ (-r_A)$

$-r_{\ A} = K\ C_A\ (1-X_A)$

Substituting $(-r_{\ A})$ into (V_R) to obtain:

V_R= volume of the reactor (dm^3) = $F_{A0}\ X_{A0}\,/\ K\ C_A\ (1-X_A)$

Length of reactor LR

As Coulson & Richardson's [16], the length L_R and the volume of reactor both given as below:

$V\ = \pi r^2\ L_R$

Where L_R = length of reactor (m)

Dimension of reactor head

L_H= length of reactor head (m) = $D_R/2$

Resident time distribution (RTD) specific time (τ)

$\tau = V_R/\ V_O$

$V_O = F_{A.O}/\ C_A$

Space -Velocity (S_v)

$S_v= 1/\ \tau$

Jacket Dimension

Height of jacket: $H_J= H_R/2$

Let the pitch (P_j) between the spiral baffles be 2dm, then numbers of Spiracles:

$N_S\ {}_=\ H_J/P_J$

Length of Jacket: $L_J = N\pi D_R$

Design for Mechanical Equations

Stirrer Design

Length of stirrer (L_{st}) $L_{\ st}$: L_{ST}= LR- C

Stirrer clearance (C): C = ½ to 1/6 D_R

Stirrer Diameter (D_{st}) : D_{st}= ½ to 1/4 D_R

Stirrer blade-width (W) : W = $1/_4$ to $1/_6\ D_{st}$

Wall baffles (B): B =$1/_{10}$ TO $1/_{12}\ D_R$

IV. SOLUTION TECHNIQUES

Parameters Flow Rate Operational

After returning to Customs Authority – Planning Department –to forecast the country's need from the CMC product, it reported that the consumption for five years was 6.956.627 Kg/year, and in other words, the annual average consumption calculated as 1391325.4 kg/year. Assumption made for product manufacturing made to be 150 ton /year:

Percentage of cellulose from lignocelluloses = 48 %

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Molecular weight of Cellulose = $C_6H_{10}O_5$ = (6X12) + (10 X1) + (5 X16)MM_A = **162 g/mol**FA_O = G/MM_A= 4444.4 / 24x60x60 = **0.0514 kmol /s**

CA_O = **0.309 k mol /cm^3**

X = mol of reacted /mol of feed

Molecular weight of CMC

C8H11O7Na = $_2$ (OH)$_2$O) CMC = (C_6H_7O

8x12+ 1x11+ 7x10 +1x23 = **242g/ mol**

The percentage of produced CMC 71 % = 720 x10 ^{3} x 71 /100 = 511.2 Kg

X= mole of reacted /mol of feed

Molecular weight of alkaline cellulose $C_6H_7O_2(OH)_2ONa$ = 184g/mol

Mol of alkaline cellulose 720*10^3/184 = **3913 k/mol**

X = 3913/444.4 =0.88

Ln k= ln A- ln E/RT

It was established, that etherification reaction is described by the equation of the -first order-

Determined activation energyE =3,088·10 ^{4} J/mol,

Pre-exponential factor A = 1,641·10 ^{3} 1/sec

Specific thermal effect: QP =1789 kJ/kg for etherification of CMC

QP =1789 kJ/kg for etherification of CMC

K = Ln 1.641.10 3 e .3088/8.314 (55+375) = 0.465* 1.0113/1000 = 0.000501 s-1

$-r_A$= k.C_nCMC - rA = KCA (1-X) = 0.000501 X 0.309 X (1- 0.88) = 1.85738 E^{-05}

Volume

V_R= FAOX / $-r_A$= 0.000501 s-1x 0.88 /1.85738 E-05= **2435.2557 dm^3**

Certainly, the outputs of the developed modeling come in Table (1), and from that it is possible to deduce the required volume according to the weight of the CMC to be produce.

Table 1: Algebraic Data calculations

x conversion	0.11	0.22	0.33	0.44	0.55	0.66	0.77	0.88
1-X	0.89	0.78	0.67	0.56	0.45	0.34	0.23	0.12
K	0.000500912	0.00050091	0.00050091	0.000500912	0.0005009	0.000500912	0.000500912	0.000500912
CA0	0.309	0.309	0.309	0.309	0.309	0.309	0.309	0.309
ra	0.0001378	0.0001207	0.0001037	0.000086678	0.00007	0.00005263	0.00003560	0.00001857
1/ra	7259.221	8282.957	9642.846	11536.976	14357.126	19002.078	28090.029	53839.222
FA0	0.0514	0.0514	0.0514	0.0514	0.0514	0.0514	0.0514	0.0514
fAo/-rA	373.124	425.744	495.642	593.001	737.956	976.707	1,443.827	2,767.336
v	41.0436	93.6637	163.5620	260.9203	405.8760	644.6265	1111.7472	2435.2557

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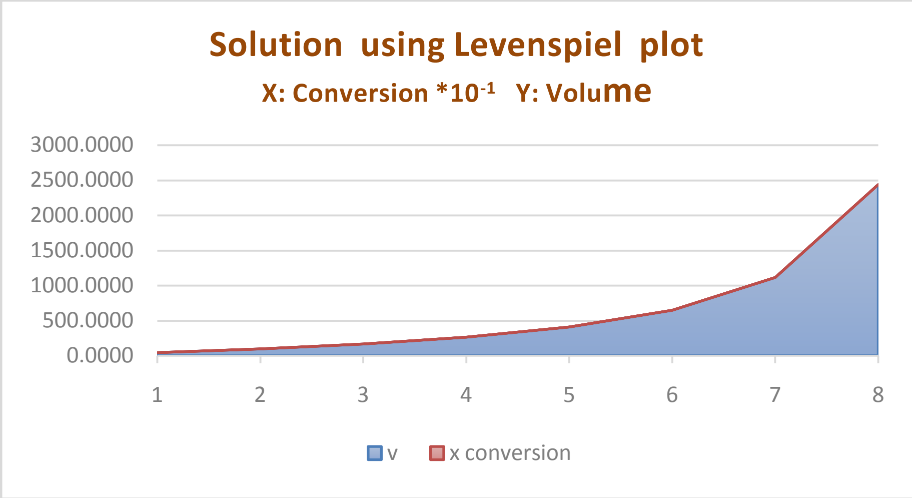

Figure 1: Plot of volume of reactor against fractional conversion

Figure 1 shows and with accordance to Leven spiel theory for substantiation, that volume of the reactor increases slowly at different reaction conversion to a point where there is a rise in the volume increase to infinity.

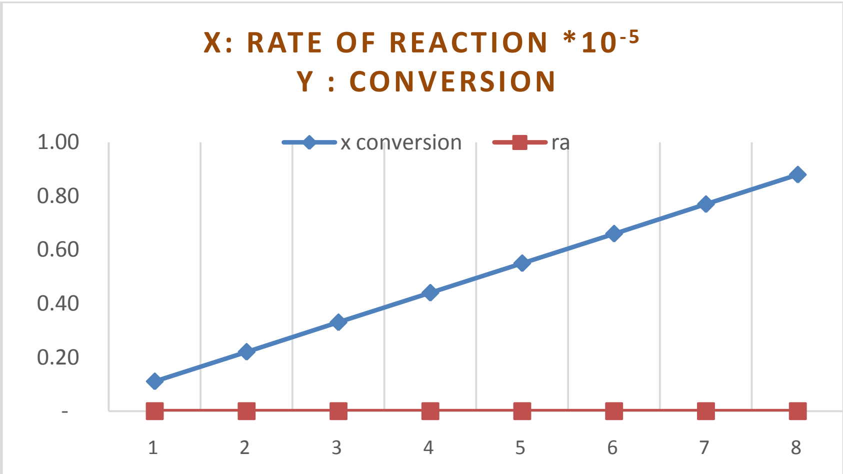

Figure 2: Plot of Rate of reaction against fractional conversion

Figure 2 shows that the rate of a reactant slowly increases upon shifting a different reaction and homogeneity with the fractional mole to a point where there is an infinitely high-volume increase.

Diameter of reactor D_R

$$D_R = \sqrt[3]{\frac{4 * V}{\pi}}$$

D_R = 14.5845(dm)

Length of reactor head

L_R= length of reactor head (dm) = 1.5 D = 1.5 * 14.5845 = 21.877 (dm)

Resident time distribution (RTD) specific time (τ):

$\tau = V_R / V_O$

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$V_O = F_{A.O} / C_A$= 0 .0514/0.309= **0.166**

$\tau = V_R / V_O$ = **14,639.96132**

Space -Velocity (S)

$S_v = 1/\tau$ = **6.83062E-05 E^{-05} h^{-1}**

Jacket Dimension

- **Height of jacket:**

HJ = LR /2 = 10.938(dm)

Assume that the pitch (P_j) between the spiral baffles be 2 dm

- **Numbers of Spiracles:**

$N_S = H_J/P_J$ **= 5.469= 6 Spiracles**

- **Length of Jacket:**

$L_J = N_s \pi D_R$ = **228.977dm**

Mechanical Design Equations

Stirrer Design

- Length of stirrer (L_{st}) L_{st}:

$L_{ST} = L_R - C$

- Stirrerclearance (C):C = ½ to 1/6 D_R C= **2.431dm**

L_{st} = 21.8768 9 - 2.4308 = **19.446dm**

- Stirrer Diameter (D_{st}) : D_{st}:½ to 1/4 D_R = **3.646dm**
- Stirrer blade - width (W) : $W = 1/_4$ to $1/_6$ D_{st} = **0.608dm**
- Wall baffles (B): $B = 1/_{10}$ TO $1/_{12}$ D_R = **1.215 dm**

V. MATERIAL SELECTION

Based on the following factors; Temperature, Allowable pressure, low thermal conductivity, PH Corrosion resistant and Heat transfer rate Austenitic stainless steel is selected as the material for constructing and fabrication of the reactor. There are more than 100 different types of stainless steel have excellent corrosion-resistance and heat-resistance properties. Carbon steel is selected for the jacket because it standard alloy in chemical industry and that after following Coulson & Richardson's [17].

Economic Benefits of CMC

CMC produced in laboratory and extracted design process from palm fronds in this Study; the process design Units to produce CMC from local raw material will undoubtedly provide the country with a large amount of hard currency that spent annually to import large quantities of CMC, which used in many purposes, including drilling fluids. In this study, refer to the Customs Authority to find out the quantities imported annually from CMC as well as their cost in hard currency during the years 2014, 2015, 2016, 2017 and 2018 as per bellow table 2.

Table 2: CMC Economical Sensitivity for Years from 2014 to 2018

Year	Net Mass-kg	Custvall- USD
2014	1469404.00	2,490,417.90
2015	1914442.00	3,906,194.74
2016	854912.00	1,544,618.66
2017	1323332.00	2,130,923.13
2018	1394537.00	1,572,685.3

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VI. CONCLUSIONS

As the purpose of investing money in chemical plant is to earn money, some means of comparing the economic performance of projects needed. The decisions usually made by comparing the capital and operating costs. For small projects, the study generated tools for utilizing Cellulose Biomasses Polymers from palm frond Fabricating Unit Design to produce 150 tons/year of carboxymethylcellulose with volume equal 2435.26 dcm3.The advantage of this work is, the conversion of plant waste materials into useful products would introduce alternative several of socio-economic materials, especially the palm trees available all over and it is frond considered waste. Therefore, after the conversation of the cellulose, which extracted from palm frond into CMC, the synthesis CMC would be more economical than the commercial one, which considered being high cost. Actually, the synthesis CMC used for many beneficial applications, for example the effect of the rheological properties of drilling fluid improved, also the viscosity of drilling fluid increased.

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