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EQUILIBRIUM AND KINETIC STUDIES OF THE DEHYDRATION OF ETHYL ALCOHOL-WATER MIXTURE VIA LOCALLY SYNTHESIZED ACTIVATED STARCH

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ABSTRACT

Batch kinetic process was employed in the evaluation of the dehydration of ethyl alcohol-water mixture via locally synthesized activated starch from cassava and maize. The equilibrium data generated were analyzed using adsorption isotherm and kinetic mechanism model equations. Results showed that the optimum concentration of 74.07% was obtained when 4g of modified cassava starch was contacted with 70ml of ethyl alcohol of initial concentration 69.72% at a contact time of 20min where the volume of ethyl alcohol recovered was 65ml. The maize starch could only achieve 72% optimum concentration when same 4g of modified maize starch was used with 70ml of ethyl alcohol with an initial concentration of 68.15%, at a contact time of 20 min where 60 ml of ethyl alcohol was recovered. The adsorption isotherm was studied using two well-known models; Langmuir and Freundlich. Although results showed that the adsorption process obeyed both models, however, the Freundlich model gave a better fit with an R^2 *value of 0.984 as against 0.974. The kinetic study on the other hand showed that the adsorption process obeyed the pseudo-second order.*

Keywords: Batch process, dehydration, ethyl alcohol, water, activated starch, Langmuir, Freundlich, isotherm

INTRODUCTION

Ethyl alcohol is an important industrial chemical which is majorly used as a solvent in the synthesis of other organic chemicals, and as an additive to automotive gasoline. Ethyl alcohol is a renewable fuel since it is obtained from biomass and burns more cleanly and completely than gasoline or diesel fuel (Diaz, 2010).The importance of ethanol as fuel and for other industrial uses has resulted in the need to produce it in a large quantity with particular reference to dehydration, (Okafor et al., 2014), and as such, a lot of research efforts continue to highlight on the improvement of the dehydration of the ethyl alcohol-water mixtures. Suffice to emphasize that concern on the global environmental protection as well as the depletion of the conventional hydrocarbon fuel supplies, have necessitated researchers to seriously look into alternative fuels such as hydro, wind, biofuels, solar and geothermal energy (Kumar et al., 2010), Amongst these alternatives, biofuels have drawn more attention (Okewale et al., 2015). Ethyl alcohol is commonly used as a fuel itself or an additive that helps improve the octane number and combustibility of gasoline (Diaz, 2010).One of the energy-efficient methods widely used for ethyl alcohol dehydration to improve its quality in terms of concentration is adsorption process. The adsorption is often carried out using starchy adsorbent, due to its numerous benefits as starch and its derivatives represent a cheap and environmentally safe source of raw material for the preparation of low-cost adsorbents (Zhang et al., 2005 &2008). Besides, starch as a biopolymer represents an interesting alternative as an adsorbent because it is an abundant, renewable and biodegradable raw material. It is interesting to note that starch, cellulose, hemicelluloses and other starch based materials have affinity for water and are able to be regenerated at temperature of 80°C and lower (Carmo and Gubulin, 1997). It has been shown that ethyl alcohol dehydration by adsorption requires far less energy than the conventional azeotropic distillation. Generally, adsorption with biomass adsorbent is less-energy consuming than adsorption with other adsorbents (Chontira and Panarat, 2010). Biomass materials that have been investigated and found to be viable adsorbents include cassava starch, corn grits, potato starch, ligno-cellulosic, amylose, and corn starch (Mya, 2011).The process and mechanism of separating ethyl alcohol using adsorption from a feed mixtures containing ethyl alcohol water, involves the mixture being contacted with the adsorbent, where the water is more selectively adsorbed and retained by the adsorbent while ethyl alcohol is relatively un-adsorbed, and removed from the interstitial void spaces between the particles of the adsorbent and the surface of the adsorbent.

Adsorption isotherm is basically important to describe how solutes interact with adsorbents whose parameters express the surface properties and affinity of the sorbent, at a fixed temperature and is critical in optimizing the use of adsorbents (Tan and Hameed, 2010).

Starch based adsorbents adsorb water by forming hydrogen bond between the hydroxyl groups (-OH) of the glucose unit and the water molecules (Beery and Ladisch, 2001), also water is by nature more polar than organic compounds such as alcohols. However, it interacts with hydroxyl groups of glucose containing adsorbent with higher strength and faster rate. Consequently, aqueous alcohol solution passing through bed of starchy or cellulose material would improve the concentration of ethyl alcohol in the solution (Okewale et al., 2011). Advantages of these starch based adsorbents in dehydration of ethyl alcohol-water mixtures include; non-toxicity, availability, biodegradable and renewability (Okewale et al., 2011). Corn starch is versatile, easily modified, and are of many uses in industry such as adhesives, in paper products as an anti-sticking agent and textile manufacturing. It has medical uses as well, such as to supply glucose for people with glycogen storage disease (Gremset al., 1990). *Zea mays* (derived from Spanish), commonly referred to as maize, belongs to a member of the grass family *Poaceae (Scott and Emery, 2016).* It is also known as corn (North American and Australian English), and it is a cereal grain first domesticated by indigenous people in Southern Mexico about 10,000 years ago (Benz, 2001). Suffice to mention that Maize (corn) starch is the starch that is derived from corn grains. The starch is obtained from the endosperm of the kernel. Corn starch is a common food ingredient, often used to thicken sauces or soups, and to make corn syrup and other sugars (James, 2018).

The major chemical component of the maize kernel is starch, which offers between 72 to 73 percent of the kernel weight. The starch in maize consists of two glucose polymers; amylose, as essentially linear molecule, making up 25 - 30 percent of starch and amylopectin, which is a branched form molecule, that makes up 70 - 75 percent of starch (Merriam, 2016).

(a). Chemistry of Cassava and Maize Starch

*Manihot*esculenta, commonly referred to as cassava, is a known woody shrub, of South America origin, which belongs to the spurge family of the Euphorbiaceae and, after rice and maize, it is ranked as the third-largest source of food carbohydrates in the tropics (Fargette et al., 1996). Cassava is starchy, and the cells of the root tubers contain starch grains (leucoplasts) from which the starch is extracted from the cassava tuber. In order to extract the starch, the cassava tubers are first crushed, where the starch grains are released from the destroyed cells. Cassava starch being a refined starch contains minimal amount of protein and fat (Fauquet and Fargette, 1999) as 100 g of cassava itself contains protein (1.4 g) and fat (0.3 g). Cassava starch contains approximately 800 ppm phosphate bound to the starch, which increases the viscosity of starch and thus gives the solution a slightly anionic character, with a low gelatinization temperature of approximately 60°C and high swelling power. These special properties make it useful in food and technical applications as wallpaper adhesive, paper coating and sizing, and for textile finishing and textile sizing, (Bemiller and Whistler, 2009).

This study therefore, focuses on the use of activated starch derived from cassava and maize for the dehydration of ethyl alcohol in order to ascertain which of these sources of starch enhanced the dehydration process better as well as to evaluate the models for the adsorption process.

MATERIALS AND METHODS

Materials and equipment: All the chemicals used in this study are of analytical grade (> 98 %). They include ethyl alcohol, sodium hypochlorite (NaOCl), sodium hydroxide (NaOH), distilled water, ionized water, hydrochloric acid (HCl). They were all obtained from Pyrex laboratory limited, Warri, Nigeria, Delta State. Others are; Blender, grater, filter paper, Oven dryer, Sieve (210 mesh size), chemical weighing balance (with accuracy of 0.01), conical flask (250ml), muffle furnace, pH meter, measuring cylinder, aluminum foil, refractometer, petri dish, cassava starch, maize starch.

(a). Sample collection

Cassavas and maize that were used in this work were obtained at a local market in Ughelli, Delta State, Nigeria.

(b). Cassava starch extraction

Cassava roots (1kg) were peeled manually with stainless steel knife, washed with 5 L of potable water to remove adhering soil and then grated using a grating machine. After grinding, the mash was submerged in 2 L of potable water and sieving was done using a muslin cloth into a fresh bowl of potable water. The extract was left to settle for 4hrs. After settling, the supernatant was disposed, and fresh water (2 L) was added to the sediment. The washing procedure was repeated five times until a clean white starch was obtained. The slurry was pressed in a muslin cloth to remove excess water and the starch sample was sun dried for a total period of 24hrs, in a batch of 6hrs a day. Dried samples were sieved and packaged in polythene can and stored at room temperature.

(c). Maize starch extraction

Maize grains of (2 tin cups) were measured into a 1000cm^3 conical flask, followed by the addition of warm water to cover the maize. The covered maize was allowed to rest for 12hrs to enable it absorb the water and become soft. It was later removed from the water and properly rinsed again with potable water. Thereafter the maize was poured into a blender, and water was added to cover the maize in the blender before blending to a smooth texture. After blending the maize, a very thick paste was recovered. This was followed by the addition of little water to mix, in order to have a runny consistency that will hasten the sieving process. The sieving was done using a muslin cloth into a fresh bowl of potable water. The extract was left to settle for 4hrs. After settling, supernatant was disposed and fresh water (75cl) was added to the sediment. The washing procedure was repeated five times until a clean white starch was obtained. Starch slurry was pressed in a muslin cloth to remove excess water and the starch sample was sun dried for a total period of 24hrs, in a batch of 6hrs a day. Dried sample was sieved and packaged in a polythene can and stored at room temperature.

(d). Proximate Analysis

Proximate analysis is the determination of moisture, volatile matter, fixed carbon, and ash content by prescribed methods (MCE, 2020). The proximate analyses were carried out before and after the activation of the starch, using the following procedures:

(e). Moisture content determination

Thermal drying method was used in the determination of moisture content of the samples. The cassava and maize starch (3g each) were weighed and placed in clean, dried, and weighed petri dishes. The petri dishes were placed in an oven at 105°C for 20 min to remove any moisture content (MCE, 2020).The difference between the initial and final mass of the starch represents the moisture content. The percentage moisture content (%) was computed according to equation (1):

Moisture % =

$$\frac{\text{initial weight} - \text{loss in weight on drying (g)}}{\text{initial weight (g)}} \times 100 \quad (1)$$

(f). Volatile matter determination

Volatile matter content was determined by heating 3g of cassava and maize starch at a temperature of 600˚C for ten minutes in the absence of air. The corresponding weight difference was reported on percentage basis. The volatile matter was calculated using equation (2) below:

Volatile (%) =

$$\frac{weight\ of\ volatile\ component\ (g)}{oven\ dry\ weight\ (g)} \times\ 100 \quad (2)$$

(g). Ash content

For ash content determination, crucibles were preheated to about 105°C in an oven for 20 min then cooled in a desiccator, and weighed. 3g each of cassava and maize sample was transferred into the crucibles and weighed. The crucibles containing the samples were then placed in the furnace and the temperature was allowed to rise to 700°C for about 1hr 30 min and allowed to cool in a desiccator to room temperature and

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reweighed. The corresponding weight difference was reported on percentage basis. The ash content was calculated using equation (3):

$$\text{Ash } (\%) = \frac{weight\ of\ ash\ component\ (g)}{oven\ dry\ weight\ (g)} \times 100 \quad (3)$$

(h). Fixed carbon content

The fixed carbon content was derived by subtracting from 100; the percentage of moisture, volatile matter and ash content. Thus,

Fixed Carbon (FC) = 100 – (%moisture + % volatile matter + % ash) (4)

(i). Activation / Modification of Cassava and Maize Starch

The unmodified cassava and maize starch (60g each) were weighed into a glass beaker separately with an addition of 150ml distilled water into both beakers. The pH of the slurry was 8.0 and 8.5 respectively for cassava and maize. 5g of chlorine was added to the slurry slowly for 10 min with constant stirring until a pH of 10.8 and 11.0 respectively was obtained and the resulting solution was allowed to stand for one hour. Finally, the pH of the mixture was adjusted to 7.0 and 8.0 respectively using 5ml HCl. The slurry was filtered through filter paper respectively. The residue was then dried in an oven at 50^{o}C for one hr, and after allowing to cool, it was packed in an aluminum foil and stored in a desiccator.

(j). Calibration Curve

To determine the concentration of ethyl alcohol solution before adsorption, refractive index calibration curve was prepared using ethyl alcohol concentration, ranging from 20 - 80% using MS excel.

(k) Stepwise Treatment Process

A stepwise treatment process was employed to improve the quality concentration of the ethyl alcohol solution. 10g each of modified cassava and maize starch were weighed on a chemical weighing balance and placed in different conical flasks. 100ml of ethyl alcohol was measured separately and added to the 10g of modified cassava and maize starch in different conical flasks. The flasks containing the solutions and adsorbent were intimately mixed and allowed to stand for 1hr. Each solution was filtered and the refractive index of the different filtrates was checked using a refractometer. The final concentration of the corresponding refractive index was then determined. The procedure was repeated with varying ethyl alcohol concentrations (%): 60.00, 63.08, 68.15, 69.72 and 74.07 %

(l). Adsorption Isotherm

Study of the adsorption isotherm was carried out on two well-known isotherms: namely Langmuir and Freundlich models. The Langmuir model is predicated on adsorption occurring on a homogenous adsorbent surface of identical sites, which are equally available and energetically equivalent. It is known to be used successfully in many monolayer adsorption processes (Allen et al.,(1988). It is expressed as:

$$q_e = \frac{q_m K_L C_e}{1 + k_L C_e} \quad (5)$$

where, C_e = equilibrium concentration of the sorbate solution; q_e = equilibrium adsorption capacity; q_m= maximum equilibrium adsorption capacity; K_L = energy of adsorption

The Freundlich isotherm on the other hand is based on the assumption that adsorption occurs on heterogeneous surfaces at sites with different energy of adsorption and with non-identical adsorption sites that are not always available (Walker et al., (2003) and it is expressed as:

$$q_e = K_f C_e^{\frac{1}{n}} \qquad (6)$$

where, K_f = Freundlich constant related to sorption capacity; $\frac{1}{n}$ = Freundlich constant related to the intensity of adsorption; C_e = starch concentration at equilibrium state.

(m). Kinetic Study

For the kinetic test, aqueous samples were taken from the 90wt% concentration of ethanol water mixtures that circulate continuously in a closed loop through a packed bed of adsorbents particle at regular time intervals using filtered syringe and thereafter, the end concentration of the sample was determined using refractometry method. The amount of water adsorbed was obtained using the formula (Mya, 2011);

$$q_e = \frac{\mathrm{ML}}{\frac{\mathrm{MA(Cf - Cl)}}{\mathrm{Cf}}} \qquad (7)$$

where, q_e = the amount of water adsorbed per unit mass of adsorbent (g H_2O/g adsorbent). C_f and C_l = final mass fraction of ethanol in solution and initial mass fraction of ethanol in solution (wt %), M_L = mass of liquid phase, (g), and M_A = mass of the adsorbent used (g). The kinetics data were fitted using

$$\frac{qe}{ce} = K_L Q_m - K_L q_e \qquad (8)$$

$$\text{and, } \frac{t}{qt} = \frac{1}{kqe2} + \frac{1}{qe}t \qquad (9)$$

RESULTS AND DISCUSSION

(a). Proximate Analyses

The results of the proximate analyses for both cassava and maize starch are presented in Table 1. The moisture contents for cassava starch (CS) and maize starch (MS) before and after activation were as low as 4.53% and 7.90%, and 3.50% and 4.68% respectively, indicating good starch. The volatile matter values were 97.00% and 99.65% for cassava starch and 94.00 and 99.28% for maize starch. The results showed that the activation process increased the volatile matter of both cassava and maize starch. The high ash content values for CS (95% & 93%) and MS (91% & 89%) is an indication of the activated starch having low organic content and high fixed carbon Kosha and Amit, 2018) with a positive effect of increasing the efficiency of the activated starch and consequently that of the recovered ethyl alcohol.

Table 1: Comparative proximate analysis of cassava and maize starch before and after activation.

Component (%)	**Before Activation**		**After Activation**	
	Cassava starch	**Maize starch**	**Cassava starch**	**Maize starch**
Moisture content	3.50	4.68	4.53	7.90
Volatile matter	97.00	94.00	99.65	99.28
Ash content	93.00	89.00	95.00	91.60
Fixed carbon	93.50	87.68	99.18	98.78

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(b). Stepwise Treatment Process

A step wise treatment process was employed to improve the quality concentration of the ethyl alcohol solution. A four stepwise contact treatment showed an improved quality concentration of alcohol-water mixture as shown in Tables 2 and 3, where cassava starch exhibited a higher improved quality over the maize starch, as an optimum concentration of 74.07% was obtained when 4g of modified cassava starch was contacted with 70ml of ethyl alcohol of initial concentration of 69.72% of a contact time of 20minwhere the volume of ethyl alcohol recovered was 65ml. The maize starch could only achieve 72% optimum concentration when same 4g of modified maize starch was used with 70ml of ethyl alcohol with an initial concentration of 68.15%, at a contact time of 20min and 60ml of ethyl alcohol was recovered.

Table 2: Stepwise treatment process of ethyl alcohol dehydration using modified cassava starch.

Time (mins)	Volume of Ethylalcohol used (ml)	Initial conc. (%) of alcohol	Amount of starch used (g)	Volume of ethylalcohol recovered (ml) after dehydration	Final conc. (%) of alcohol after dehydration	Refractive index	Stage
20	100	60.00	10	90	63.08	1.3599	1
20	90	63.08	8	85	68.15	1.3611	2
20	85	68.15	6	70	69.72	1.3625	3
20	70	69.72	4	65	74.07	1.3642	4
20	65	74.07	2	60	74.07	1.3642	5

Table 3: Stepwise treatment process of ethyl alcohol dehydration using modified maize starch.

Time (min)	Volume of ethyl alcohol used (ml)	Initial conc. (%) of alcohol	Amount of starch used (g)	Volume of ethyl alcohol recovered (ml) after dehydration	Final conc. (%) of alcohol after dehydration	Refractive index	Stage
20	100	60.00	10	95.00	62.00	1.3599	1
20	95	62.00	8	87.00	66.05	1.3611	2
20	87	66.05	6	78.00	68.15	1.3625	3
20	78	68.15	4	60.00	72.00	1.3642	4
20	60	72.00	2	57.00	72.00	1.3642	5

(c). Adsorption Isotherms

Both Langmuir and Freundlich adsorption isotherms were tried to correlate the best fit for the ethyl alcohol adsorption on the activated starch. The results presented in Figures 1 and 2 showed higher value of correlation coefficient of Freundlich adsorption isotherm (0.9837) of ethyl alcohol over the Langmuir isotherm (0.9740). K_L was determined to be 4.88.

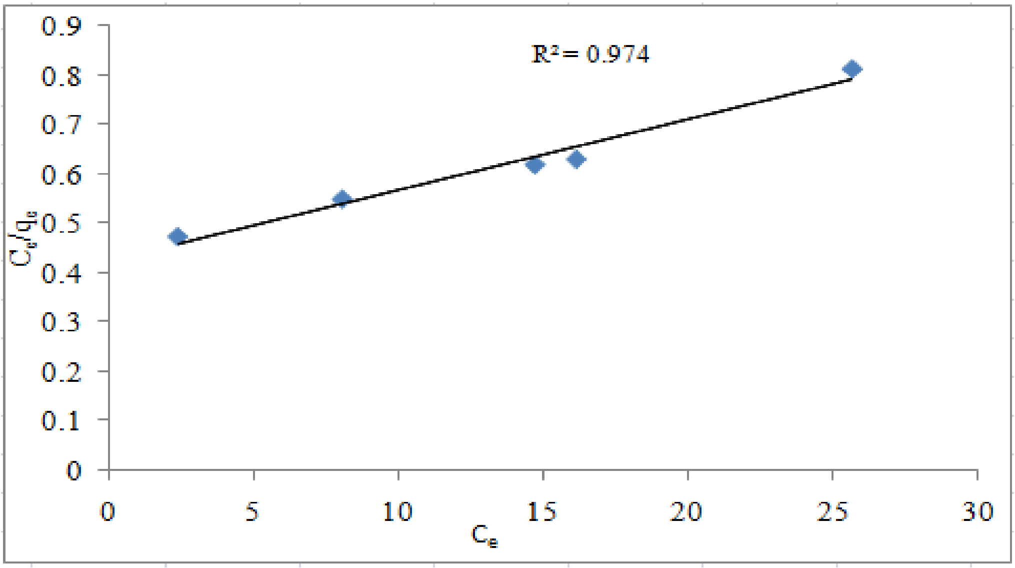

Figure 1: Langmuir adsorption model of ethyl alcohol water mixture on modified cassava starch

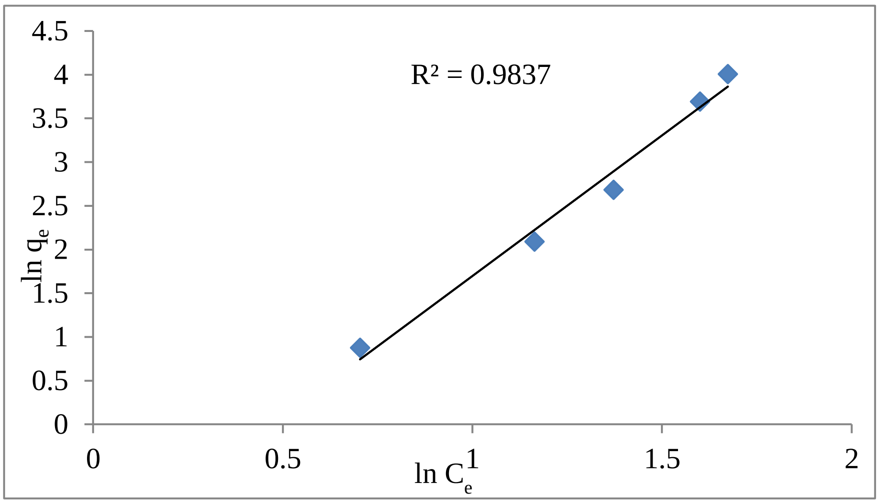

Figure 2: Freundlich adsorption model of ethyl alcohol water mixture on modified cassava starch

(d). Adsorption Kinetic

The kinetic study involved three variable concentrations according to Equation (7); ethanol-water mixture and starch. However, one of the variables (starch) did not change over time, since it was present in excess amount, whereas the ethanol varies with time. Hence, the experimental result obtained as shown in Figure 3 conformed to a pseudo-second order, as the high R^2 value (0.968) obtained from the pseudo-second order model indicated the applicability of the pseudo-second order kinetic model to describe the adsorption process of water uptake on the starch-based adsorbent. This indicates that the overall rate of the adsorption process was controlled by chemisorption which involved valence forces through sharing or exchange of electrons between the sorbent and the sorbate (Ho and McKay., (1999).

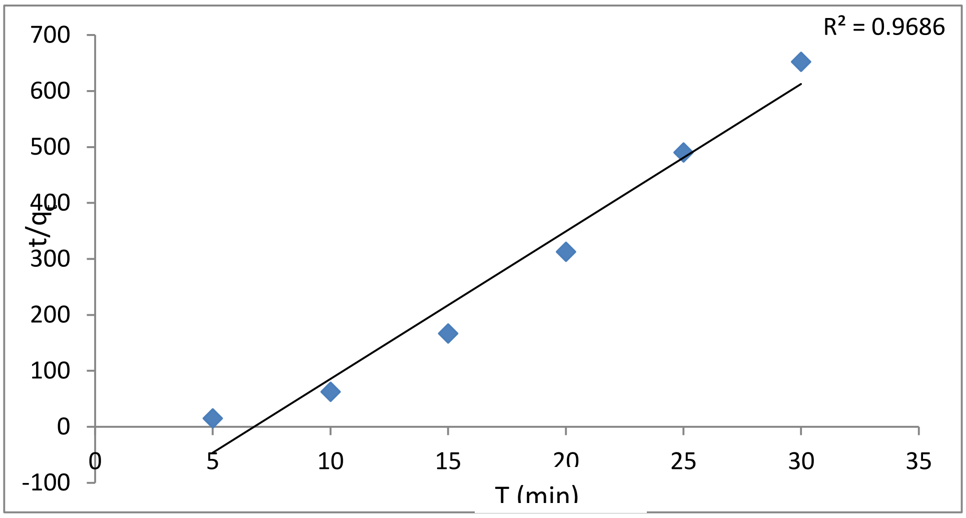

Figure 3: Pseudo-second order of water removal from ethyl alcohol using activated cassava starch.

CONCLUSION

The cassava and maize starch modified with sodium hypochlorite, hydrochloric acid and sodium hydroxide was found as a promising adsorbent for the dehydration of ethyl alcohol water mixture. The four stepwise contact treatment improved the quality (concentration) of the ethyl alcohol-water mixture via dehydration, as the concentration was raised from 69.72 and 68.15% to 74.00, and 72.00% respectively, for both cassava starch and maize starch. From the results obtained, modified cassava starch was found to adsorb more water from the ethyl alcohol- mixture than modified maize starch, with a better fit of Freundlich model correlation over Langmuir adsorption isotherm, as well as obeying a pseudo-second order model equation.

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