

Microcontroller Based Automated Reactor for Esterification of Lactic Acid: MATLAB Simulation

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ABSTRACT: This paper deals with the esterification of lactic acid using sulfonated carbon catalyst in a newly designed Microcontroller-Based Automated Reactor (MBAR) and the simulation of process parameters using MATLAB programming. The reactor was accompanied by a moisture sensor, temperature sensor, and solenoid valves in the embedded system. The study of the effect of process parameters such as silica gel weight, hot air temperature, molar ratio, and conversion of lactic acid on the removal of water, generated during esterification reaction, was performed. Water removal by adsorption using silica gel at each stage of conversion was estimated experimentally as well as with the help of developed, simulated linear equations, using MATLAB. The experimental and MATLAB results were compared and found in close vicinity. The simulation results revealed that increased water removal is achieved with increasing conversion and molar ratio. The results also validated that increasing the reaction temperature increases the conversion tremendously with a rapid decrease in hot air flow requirement. The uniqueness of the newly designed reactor is that the silica bed is operated in rotation in such a way that when one is in operation another is regenerated during its idle time.

KEYWORDS: Lactic Acid; Esterification; Simulation; Catalysis; MATLAB; MBAR.

INTRODUCTION

In most of the esterification processes, yield is normally limited by thermodynamic equilibrium when the reaction takes place in the presence of homogenous catalysts [1]. Though homogeneous acid catalyst enhances the kinetics, on the other hand, it causes erosion to the process

equipment [2]. Esters of lactic acid obtained by esterification are used in the chemical industry because it is less toxic as well as biodegradable [3]. High conversion of lactic acid (about 82 %) using an inexpensive catalyst even at low loading has been reported by another

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researchers [4]. However, considerable enhancement of lactic acid conversion using pervaporation can be increased to 86% using a PVA-PES membrane [5]. Nowadays, monitoring of enhancement of esterification using reactive distillation accompanied by simulation and modeling helps to compare and control the process parameters [6]. MATLAB is technical computing and efficient software that can handle numerous complex data, thereby reducing the workload of the researchers to a great extent [7]. Usually, MATLAB is more helpful in dealing with equations that have no analytical solution that may become difficult to compare experimental results with theory [8].

Modeling and simulation technologies are widely accepted due to their flexible adeptness to quick consequences in the demand and supply of products [9]. In many processes, time dependant variable causes complexity which is well handled using computational modeling [10]. Not only this but also the reactions involving multistep reversible series-parallel elementary reactions are simulated using MATLAB tools [11]. Many reactions occurring in the field of esterification processes which can take the form of equations having a combination of ordinary differential equations and algebraic equations consisting of a complex system can be solved by MATLAB simulation [12]. Parameter tuning and signal visualization in the real-time process removes difficulties in the real process by using SIMULINK, which is used to simulate dynamic systems. SIMULINK's external mode and real-time windows target software can be integrated to achieve the solution [13]. The simulated model assists the study of dynamic characteristics of biochemical reactors. It also helps to develop the design of the fluidized bed to overcome the instability of the operation and improve the uniformity of products [20, 21].

The novelty of the present research includes the fabrication of a reactor for the enhancement of an esterification process with simultaneous removal of water generated during the reaction. The reactor was further developed to automate the apparatus using an electronic process control system in such a way as to speed up the process of esterification that can overcome the drawbacks of the pervaporation process. MATLAB programming was used to simulate the process of water removal generated during the esterification reaction. Some researchers [14, 15] reported that certain desiccants absorb water up to 5%

by weight from the alcohol-water mixture. Researchers also reported more adsorption capacity of water vapor than liquid water. Hence, adsorption methodology was used to remove water from esterification reaction products. Several model equations are simulated in MATLAB to investigate the effects of various process parameters on the removal of water.

EXPERIMENTAL SECTION

Development of an automated embedded-assisted system for esterification reaction

A Microcontroller-Based Automated Reactor (MBAR) was used for the esterification reaction of lactic acid and iso-butanol shown in Fig. 1. The reaction was conducted in 250 mL three-necked conical flask. The heat was supplied through a heater cum magnetic stirrer having a capacity 0.5 kW. Silica gel was used to remove the moisture by adsorption from the reaction mixture which was placed in cylindrical vessels of 5 cm long and 3.8 cm in diameter. The vessel was connected to the reactor by a U-shaped tube of 1.2 cm diameter for separation to occur. Moisture and temperature sensors were submerged in silica gel for detection of moisture and temperature respectively.

The whole apparatus as well as the process of esterification reaction were programmed and automated using an embedded system. Arduino board (using ATmega-328), relays, and LCD display were used to control the parameters. The interfacing of the embedded system with all the individual components, valves, and sensors forms a circuit. The sensor monitors and provides feedback for automation, whereas relays interface and operates solenoid valves. Temperature and moisture levels were displayed on a 16 x 2 alpha-numeric LCD display. Hot air flow measurement was accomplished with the help of vane type anemometer (0 – 45 m/s).

Working on newly designed MBAR

As shown in Fig. 1(b), initially all the solenoid valves (4, 5, 6, and 7) were in the closed position. Heater cum magnetic stirrer (at 400 rpm) was turned on to heat the reaction mixture (lactic acid, iso-butanol, and catalyst) uniformly in the flask till it reaches 90 °C. When the solution in the flask becomes hot and a reaction takes place, water is formed as a by-product and it gets evaporated. The vapor was directed towards silica bed-1 (8) by opening

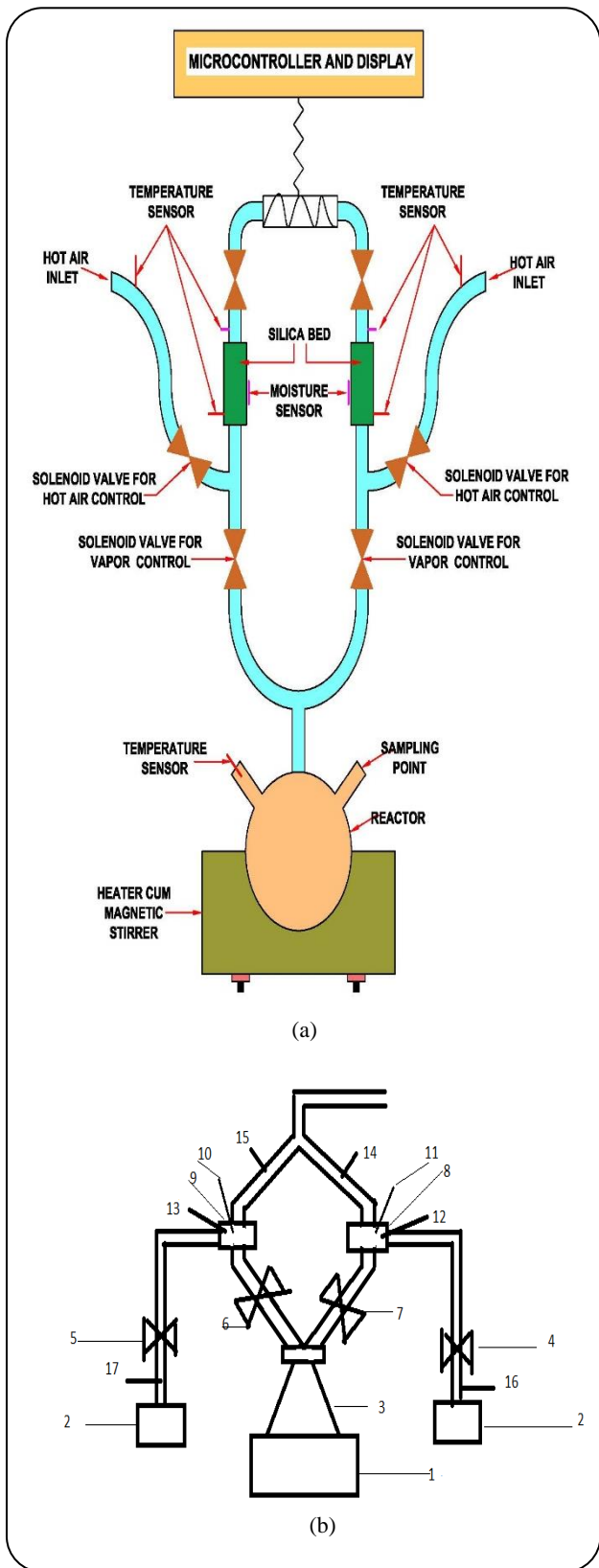


Fig. 1: (a) Experimental setup of MBAR, (b) Working of MBAR.

solenoid valve (7). Vapor gets absorbed by the silica bed-1 till the bed becomes saturated. Moisture sensors (10 and 11) and temperature sensors (12 and 13) were calibrated for the saturation point of silica gel in the silica beds and displayed on LCD at runtime. Once the silica bed-1 (8) gets saturated, the moisture sensor (11) signals the microcontroller which in turn closes the solenoid valve (7). Then the silica bed-1 could come in contact with cool air. Due to cooling, separation of iso-butanol and water takes place, where water vapor is absorbed by the silica bed, increasing its moisture level. When the moisture in silica bed-1 was saturated, iso-butanol gets separated by evaporation as it is not absorbed by the silica bed-1. Valve (7) opens for 5 seconds and closes again to separate iso-butanol, which returns to the flask. Once valve (7) closes, the microcontroller opens the hot air-controlled solenoid valve (4). The air dries the silica bed-1(8) while moisture sensor (10) and temperature sensor (12) monitor it. As valve (7) closes, valve (6) opens, and reaction vapor is directed to silica bed-2 (9). Once the silica bed-1 gets dried completely, valve (4) closes, and the air flow stops.

Display of temperature and moisture levels on LCD screen helps to take out a sample from the reaction mixture after every 30 minutes. The sample mixture was then analyzed for conversion through titration. The vapor directed to silica bed-2 was absorbed by the silica bed-2. At the same time, silica bed-1 in the other arm is in drying mode. The saturation is monitored by moisture sensor (10) and temperature sensor (13). When silica bed-2 saturates, the moisture sensor (10) signals the microcontroller which then closes the valve (6) to stop the further flow of vapors into the silica bed-2. Bed-2 was then allowed to come in contact with cool air. Again, due to cooling, separation of iso-butanol and water takes place by evaporation, where the moisture level of bed-2, increases. The moisture sensor (10) and temperature sensor (13) monitor the silica bed 2, which was displayed on LCD at runtime. When the silica bed-2 gets saturated, iso-butanol gets separated as it is not absorbed by the silica bed. Valve (6) opens for 5 seconds and closes again to separate iso-butanol which returns to the flask. Once valve (6) closes, the microcontroller opens the hot air solenoid valve (5) to dry the silica bed 2, after which valve (5) closes. The valve (6) now opens, and the process was repeated till complete evaporation of water takes place. The LCD display readings were noted down and the titrimetric analysis

of reaction, samples were repeated at least twice within the repeatability of $\pm 5\%$.

MATLAB Simulation

The esterification of lactic acid with iso-butanol in the presence of a sulfonated carbon catalyst was carried out in MBAR. As the reaction proceeds the water generated is adsorbed onto silica gel till it gets saturated. Equation (1) depicts the relation of water generated with an initial concentration of lactic acid, the molar ratio of iso-butanol to lactic acid as well as the conversion of lactic acid. Various equations were modeled in the adsorption process using silica desiccant. These equations were easily coded in MATLAB program and simulated for a variety of process variables and graphs were also obtained from the simulation. Equations (2), (3), and (4) were modeled for 20, 30, and 40g of silica gel respectively. The water absorption rate in a thicker bed is reported to be higher since the moisture diffuses into the bed having lower overall resistance [16].

$$\text{Mass of water outlet, } Q_w = \quad (1)$$

$$\left((N_{1a0} \times X_{1a}) \times (M - N_{1a0} \times X_{1a}) \right) \times 18$$

$$M\% = \frac{10000 Q_w}{\left((W_s + Q_w) \left[100 - 89.31 \exp\left(\frac{Q_w}{-1.78} \right) + 9.02 \right] \right)} \quad (2)$$

$$M\% = \frac{10000 Q_w}{\left((W_s + Q_w) \left[100 - 83.12 \exp\left(\frac{Q_w}{-1} \right) + 17 \right] \right)} \quad (3)$$

$$M\% = \frac{10000 Q_w}{\left((W_s + Q_w) \left[100 - 87.13 \exp\left(\frac{Q_w}{-2.54} \right) + 8.23 \right] \right)} \quad (4)$$

Air flow rate,

$$G_s = \frac{q_s (1 - X_2) (X_1 - X_2)}{(Y_1 - Y_2)} \quad (5)$$

As soon as the moisture is absorbed in the silica bed, its corresponding analog value is displayed on the LCD screen of MBAR. Depending on the weight of silica, Equations (2), (3), and (4) can be used to determine $M\%$ which models the relation between moisture content ($M\%$)

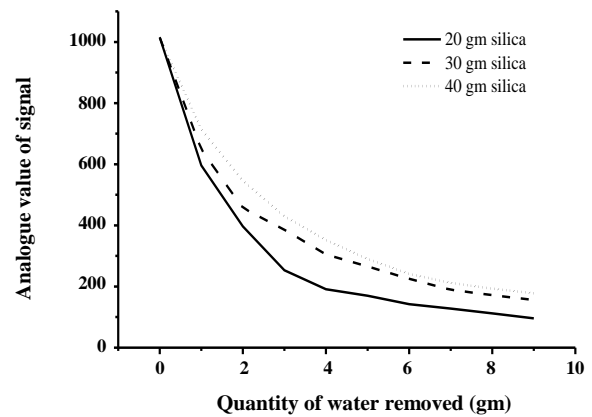


Fig. 2: Relationship of analog signals with the quantity of water removed for variable weights of silica bed.

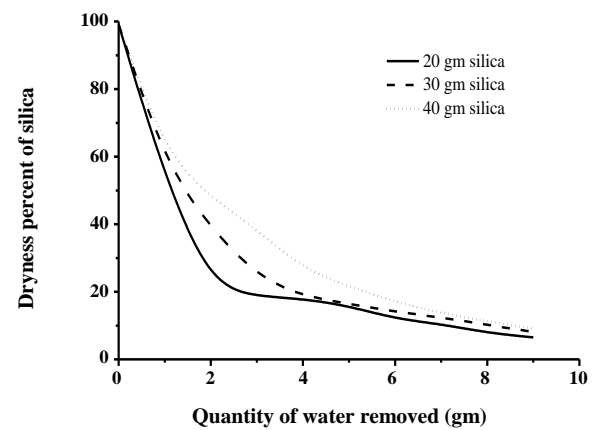


Fig. 3: Relationship of dryness percentage of silica on the quantity of water removed for variable weights of silica bed.

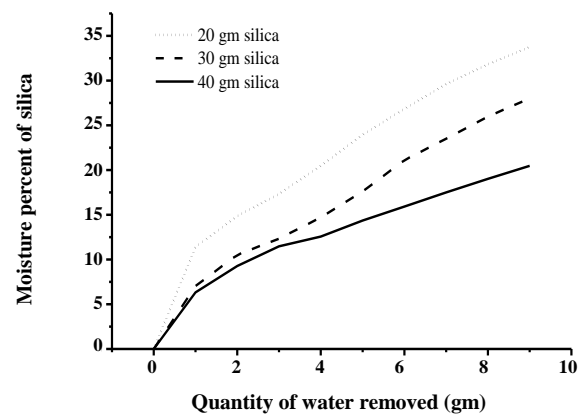


Fig. 4: Relationship of moisture percentage of silica with the quantity of water removed for variable weights of silica bed.

the weight of silica (W_s), and water removed (Q_w) equivalent to the initial moisture in silica (S_{mi}) hence, $S_{mi} = M \%$.

Based on the moisture content of both the silica beds and local humidity of incoming air, the required air flow rate, G_s can be estimated using Equation (5). However, the initial enthalpy of silica (H_{s1}) can be estimated by Equation (6) based on the initial temperature of the silica bed.

$$H_{s1} = [C_s(S_{t_i} - T_0) + X_1 C_a(S_{t_i} - T_0)] \quad (6)$$

When the silica becomes saturated by moisture absorption, it is regenerated using hot air till the final moisture content (S_{mf}) lowers to 0.1%. At this stage, the final temperature of the silica bed is also sensed, and the final enthalpy of silica (H_{s2}) is determined by Eq. (7).

$$H_{s2} = [C_s(S_{t_r} - t_0) + X_2 C_a(S_{t_r} - t_0)] \quad (7)$$

The net transport of moisture can be determined using equation (8) based on inlet and outlet enthalpies of air. This is the direct measure of moisture content in the air.

Enthalpy of inlet air,

$$H_{g2} = [1005 + (1884 Y_2)] T_{g2} + (Y_2 \lambda_a) \quad (8)$$

The absolute humidity of the available air (Y_2),

$$\frac{\text{Kg of water}}{\text{Kg of dry silica}} \quad (9)$$

Where Y_2 is taken as 0.01 (1%) for calculation purposes. Heat absorbed from hot air by saturated silica bed causes water evaporation which results in a reduction of relative humidity. This phenomenon leads to an increase in the absolute humidity of surrounding air and subsequently decreases the vapor pressure gradient and the rate of drying. The enthalpy of outlet air, H_{g1} is determined by equation (10) based on required air flow rate G_s and absolute humidity of outlet air Y_1 .

Enthalpy of outlet air,

$$H_{g1} = (1005 + 1884 Y_2) 32 + \lambda_a Y_1 \quad (10)$$

Thus, with the help of Equations (6) to (9) and the magnitude of moisture and temperature displayed by LCD on the reactor, it is possible to determine the required flow rate of hot air by using Equation (5). Equations (11) to (13) are developed to calculate the time required for conversion which ultimately gives the time for moisture removal.

RESULTS AND DISCUSSION

Effect of conversion of lactic acid on the water removal

From Fig. 5, it is observed that as the reaction proceeds, the conversion increases, likewise the amount of water in the system generated leads to hindering the forward shift [17]. Conversion is a function of time, t , and exhibits a definite relevance. The Equations (11) to (13) give the relationship at 90, 95, and 100°C respectively. These model equations developed are helpful for coding in MATLAB and thereby estimating the water removal in the esterification.

$$t = 10 \log \left\{ \frac{(X_{la} * 100) - 193.77}{(-192.17)} \right\} * (-417.62) \quad (11)$$

$$t = 10 \log \left\{ \frac{(X_{la} * 100) - 237.27}{(-231.16)} \right\} * (-557.57) \quad (12)$$

$$t = 10 \log \left\{ \frac{(X_{la} * 100) - 666.88}{(-655)} \right\} * (-1897.27) \quad (13)$$

Effect of molar ratio on the water removal

Fig. 6 shows that increasing the molar ratio, there is an increase in the conversion of lactic acid as well as the water content in the mixture [18]. MATLAB result proposes 16 to 23% water removal for the range of 2 to 3 molar ratio.

Effect of silica gel weight on the water removal

Fig. 7 clearly indicates as the conversion of lactic acid increases, the quantity of water generated decreases. Consequently, water removal decreased with the increase in the bed thickness due to suppressed moisture diffusivity, in hot convective drying [19].

Influence of hot air flow rate on water removal

Two cylindrical vessels containing silica gel are used in MBAR to operate in rotation so that when one bed is in operation, alternatively the other will be in regenerating mode. The regeneration is carried out by interacting hot air flow with beds of saturated silica gel shown in Fig. 1. In this work, the effect was also studied at selected variables keeping all other parameters constant using MATLAB programming and the results are produced in Tables 1 to 5. Table 1 reveals that for higher conversion the requirement of hot air decreases slightly. This is since more the conversion of the main product, less will be

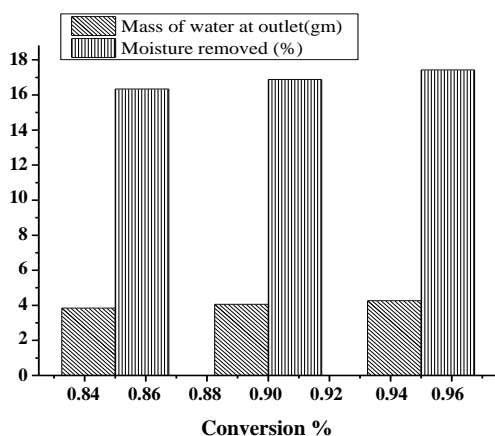


Fig. 5: Effect of conversion on the mass of water at the outlet and moisture removal.

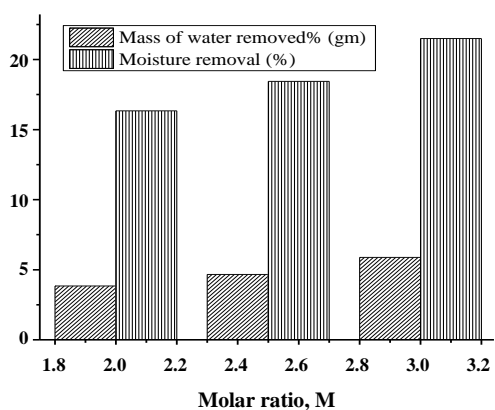


Fig. 6: Effect of molar ratio on the mass of water at outlet and moisture removal.

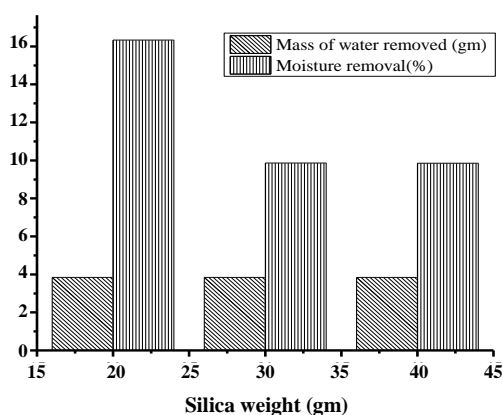


Fig. 7: Effect of silica weight on the mass of water at outlet and moisture removal.

The quantity of water generated. The heat from the hot air is transferred to the bed of silica gel by convection in absence of other heat effects. The surface temperature begins to approach the wet-bulb temperature. Under this condition, the rate of heat transfer increases and results in a higher drying rate.

Table 2 shows that by increasing the molar ratio of iso-butanol to lactic acid, there are increases in water production during the reaction. Furthermore, the conversion of lactic acid is increased by simultaneous removal of water [24]. The water absorption rates in thicker silica gel beds become higher due to the lower overall resistance offered by the silica bed for the incoming moisture, which results in higher absorption rates [18].

Table 3 shows that by increasing the weight of silica beyond 30 g, there is an increase in the bed thickness which consequently results in enlarging the regeneration time. This is due to more moisture accumulation in the bed which results in declined drying rate, hence hot air flow rate increases at a constant temperature [22]. It also indicates that the hot air requirement increases initially up to the weight of 20 g of silica bed and then decreases. This is attributable to the reason that the drying rate decreases with the increase in bed thickness due to suppressed moisture diffusivity in hot convective drying [19]. Also, a thinner bed offers lower resistance to mass transfer diffusion of water vapor than the thicker bed [24].

Table 4 shows that by increasing the hot air temperature, there is a decrease in the air flow requirement. The hot air temperature has an inverse relation with the flow rate. The slower the air moves to dry the bed, the warmer is the outlet moisture due to lesser contact of inlet hot air on the silica gel surface [23].

Table 5 shows that when the reaction temperature is increased, the conversion of lactic acid increases to suppress the formation of water due to the frequency of effective collision among the reacting species, which causes the equilibrium shifts to the product side. Hence for the same value of conversion even at high temperature, there may exist more water molecules that demand a higher hot air flow rate for its removal.

CONCLUSIONS

A newly designed MBAR reactor was used for esterification reaction between lactic acid and iso-butanol in which water removal was emphasized by its adsorption

Table 1: Effect of conversion on air flow rate.

Parameters	Values		
X_{la}	0.85	0.9	0.95
M	2	2	2
W_s	20	20	20
Th	60	60	60
T_R	90	90	90
G_s	0.102	0.101	0.099

Table 2: Effect of molar ratio on air flow rate.

Parameters	Values		
X_{la}	0.85	0.85	0.85
M	2	2.5	3
W_s	20	20	20
Th	60	60	60
T_R	90	90	90
G_s	0.102	0.129	0.171

Table 3: Effect of weight of silica gel on air flow rate.

Parameters	Values		
X_{la}	0.85	0.85	0.85
M	2	2	2
W_s	20	30	40
Th	60	60	60
T_R	90	90	90
G_s	0.102	0.079	0.103

Table 4: Effect of hot air temperature on air flow rate .

Parameters	Values		
X_{la}	0.85	0.85	0.85
M	2	2	2
W_s	20	20	20
Th	60	65	70
T_R	90	90	90
G_s	0.102	0.082	0.068

Table 5: Effect of reaction temperature on air flow rate.

Parameters	Values		
X_{la}	0.85	0.85	0.85
M	2	2	2
W_s	20	20	20
Th	60	60	60
T_R	90	95	100
G_s	0.102	0.104	0.108

using silica gel bed and its moisture level was sensed and displayed on LCD screen. The effects of all the parameters like silica gel weight, reaction temperature, conversion, and molar ratio on water removal were studied. Various equations were modeled for all the process parameters and simulated using MATLAB. It can be concluded that the requirement of hot air decreases slightly as conversion increases but increases rapidly with increasing the molar ratio of alcohol to acid. However, the flow of hot air is independent of the weight of silica gel. Moreover, simulation results confirmed that there is a decrease in hot air flow with an increase in air temperature. These results were compared and validated with the experimental results and found to be in agreement with each other closely.

Nomenclatures

X_{la}	Conversion of lactic acid
N_{la0}	Moles of Lactic acid
M	Molar ratio of iso-butanol to lactic acid
W_s	Weight of silica gel, gm
Th	Hot air temp, °C
T_R	Reaction temp, °C
G_s	Hot air flow rate, kg/h
t	Time for conversion of lactic acid, min
C_s	Specific heat of silica, J/kg°C
C_a	Specific heat of water, J/kg°C
T_{g2}	Initial temperature of air, °C
T_{g1}	Outlet temperature of air, °C
G_s	Air flow rate, kg/h
Q_w	Water entering in silica, mL
W_t	Total weight, gm
$M\%$	Moisture %
λ_a	Latent heat of air, J/kg
Y_1	Absolute humidity of outlet air
Y_2	Absolute humidity of available air

q_s	Moisture flow rate, Kg/h
H_{s2}	Final enthalpy of silica, J/kg dry silica
H_{s1}	Initial enthalpy of silica, J/kg dry silica
H_{g2}	Enthalpy of inlet air, J/kg dry air
H_{g1}	Enthalpy of outlet air, J/kg dry air
X_2	Final moisture, kg of water/kg of dry solid
X_1	Initial moisture, kg of water/kg of dry solid
St_f	Final temperature of silica, °C
St_i	Initial Temperature of silica, °C
Sm_i	Initial moisture of silica, %
Sm_f	Final moisture of silica, %
SV	Solenoid valve
SB	Silica gel bed
MS	Moisture sensor
TS	Temperature sensor

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