Development of Non-Stirred Pressure Reactor for the Production of Levulinic Acid

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Abstract

This paper presents the development and fabrication of high pressure batch reactor developed for the fractionation of lignocellulosic biomass. The availability and reliability of high pressure reactors in East African countries is very limited and expensive if imported. Therefore, a high-pressure batch reactor was developed and fabricated using SS-304 for the production of levulinic acid from Tanzanian sugarcane bagasse – a kind of lignocellulosic biomass. This high-pressure reactor was found to withhold a temperature upto $250^{\circ}C$ and a pressure of 16 bars under acidic conditions. No leakage from any part of the reactor and corrosiveness was observed during the production of levulinic acid from Tanzanian sugarcane bagasse under acidic conditions.

Keywords — *High pressure non-stirred reactor, levulinic acid, lignocellulosic biomass, biorefinery.*

I. INTRODUCTION

Industrial production of levulinic acid is one of the areas that have drawn interest of many researchers. Levulinic acid has drawn much attention in the pharmaceutical and agrochemical industries in recent years and as an ingredient in many food formulations [1]. The United States Department of Energy has identified ester of levulinic acid as a platform chemical for the production of transportation fuel [2]. Moreover, levulinic acid is a versatile building block for the synthesis of various organic compounds, biodegradable herbicides [3] and precursor in the polymer and resin industries [4,5]. Following these government promising applications. manv organizations and private companies have invested much in biomass research for the production of levulinic acid. The production of levulinic acid from biomass depends on type of biomass used as precursor, reactor parameters, and production parameter to which the production process is subjected to. One of the growing challenges towards the production of LA is the availability and reliability of the reactors. In many parts of Africa including Tanzania, getting a proper reactor for the production of levulinic acid is a serious problem. Many places in Tanzania do not sell rectors, until it is purchased from abroad, something which is very expensive.

A pressure reactor, sometimes referred to as a pressure tube, or a sealed tube, is a chemical reaction

vessel which can conduct a reaction under high pressure and elevated temperature. A pressure reactor is a special application of a pressure vessel. The pressure can be caused by the reaction itself or created by an external source, like hydrogen in catalytic transfer hydrogenation. A pressure reactor several advantages offer over the can conventional round-bottom flask. Firstly, it can conduct a reaction above the boiling point of a solvent. Secondly, the pressure can reduce the reaction volume, including the liquid phase, and in turn increase concentration and collision frequency, and accelerate a reaction. Increase in temperature can speed up the desired reaction, but also speed up the decomposition of reagents and starting materials. When the desired reaction is accelerated, competing reactions are minimized. Pressure generally enables faster reactions with cleaner reaction profiles [6].

The use of pressure reactors for research has number of challenges that requires a close observation and quality testing of the reactors. Among the challenges [7] reports fraught with difficulties severe hydraulic and mechanical problems, temperature and pressure control outages and interruptions for data acquisition purposes or other, often unexpected transients.

The availability and reliability of the reactors, in many parts of Africa including Tanzania is very limited. Getting a proper reactor for the production of platform and specialty chemicals from lignocellulosic biomass, for example levulinic acid, is a serious problem. Many places do not sell rectors, until it is purchased from abroad, something which is very expensive. Therefore, the present study is aimed at developing a non-stirred pressure batch reactor which can withhold temperature upto 250 °C and pressure of 14- 16 bars for the production of levulinic acid from Tanzanian sugarcane bagasse.

II. MATERIALS AND METHODS

A. Material of Construction

For a properly designed pressure chemical reactor, selection of materials is one of the prime conditions to be taken into account. Baddoo [7] reports the features of stainless steel reactor as a potential candidate for reactor designing. Among the features indicated, Baddoo reports that a stainless steel is a corrosion-resistant and long-lasting, making thinner and more durable structures possible. Due to its ability to resist corrosion [8] stainless steel is the most

common material used for the designing of most chemical reactors. In the present study, SS- 304 stainless steel was used for the designing of the reactor. It is also called austenite steel 304 and is composed of 0.047% C, 0.33% Si, 1.1%Mn, 0.026% P, 0.002% S, 18.1%Cr, 0.31%Mo, 9.0%Ni, 71% Fe and 2B surface finishing[9]

B. Description of the drawing

The drawing (Fig 1) is the model of the designed reactor where the numbers are explained as follows; 1- A cylindrical body of the reactor for carrying out the reaction mass. 2- Reactor thickness(4mm), 3-Thermowell elongated into the cylindrical body of the reactor which is closed at the bottom for inserting the thermometer and recording the temperature of the reaction mass. 4- Bolts for tightening the reactor in order to avoid leakage. 5- Flat Head of the reactor which is surrounded by eight bolts for tightening the reactor. 6- Nozzle used to control the flow of gas and liquid from the reactor. 7- Pressure regulator for the purpose of pressure release, it allows the release of excess vapors or drawing of samples whenever necessary. 8- Pressure gauge for recording the pressure that builds in the reactor during the course of the reaction.

III. RESULTS AND DISCUSSION

The reactor was designed on the basis of the optimization parameters for the production of levulinic acid from Tanzanian sugarcane bagasse, which are; concentration of the hydrolyzing acid, temperature of the reaction, reaction time and pressure of the reaction. A SS-304 cylindrical



stainless steel pressure reactor of inner diameter **100 mm**, depth of **200 mm** and thickness of **4 mm** was developed. The reactor's top cover is a flat stainless steel square sheet of **4mm** thickness from which the pressure gauge of **25 bars** together with a temperature probe has been fixed using gas welding. The reactor is tightened by means of eight bolts which joins it with the reactor to avoid leakage. The welding of the reactor was done by gas welding as well as electric welding by using the same stainless steel material (SS-304).

The volume of the reactor is 1730mL, the maximum temperature tested is 250° C and the maximum pressure attained is 16 bars. The maximum allowable water loading capacity[9] of the reactor is 1353.9mL or below as per the formula given below:

Sulphuric acid was used as the hydrolyzing acid at a concentration range from 0.25M to 1.5M. Temperature was varied from 180 $^{\circ}$ C to 250 $^{\circ}$ C and the reaction time was varied from 10 min to 20 min. The present reactor was used for Tanzanian sugarcane bagasse fractionation to obtain levulinic acid. The Tanzanian sugarcane bagasse was hydrolyzed using sulphuric acid at a concentration of 0.25M- 1.5M and at a temperature range from 180 $^{\circ}$ C- 250 $^{\circ}$ C with reaction time ranging from 10 min- 20 min. No leakage was observed from any part of the reactor upto a temperature of 250 $^{\circ}$ C and at pressure upto 16 bars.

Fig 1: Design of the non-stirred pressure reactor developed for the production of levulinic acid.

IV.CONCLUSIONS

A high-pressure reaction for the fractionation of sugarcane bagasse to produce levulinic acid was carried using the high-pressure reactor developed in our laboratory. The fractionation of sugarcane bagasse was carried out using dilute sulphuric acid (0.25 - 1.5)M) at a temperature range of 180°C to 250 °C and pressure between 13- 15 bars. No leakage was observed during the course of the reaction when the high-pressure reaction was carried out upto 250 °C and pressure between 13-15 bars. The production of platform and specialty chemicals from biomass is gaining interest around the world. The development of high pressure batch reactors will be of potential interest for the researcher working in the field of biomass research and government agencies of the lowincome and developing countries like Tanzania as these kinds of reactors are rarely available and becomes very expensive if imported from outside.

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REFERENCES

- [1] K. B. Efremov AA, Pervyshina GG. Production of levulinic acid from wood raw material in the presence of sulfuric acid and its salts. *Chem. Nat. Compd.* 34(2) 182–185, 1998.
- [2] Bozell JJ, Petersen GR Technology development for the production of biobased products from biorefinery carbohydrates; the US Department of Energy's 'Top 10 revisited'. Green Chemistry 12, 539–554, 2010.
- [3] L.Moens. Synthesis of acid addition salts of deltaaminolevulinic acid from 5-bromo levulinic acid ester. US patent 5907058, 1999.
- [4] A.R. Bader, A.D. Kontowicz, c, c-Bis-(p-hydroxyphenyl)valeric acid, J. Am. Chem. Soc. 76 (17) 4465–4466, 1954.
- [5] R.E. Holmen, Derivatives of Bisphenolic Substituted Carboxylic Acids, US Patent 3471,554, 1969.
- [6] [6] M. A. Herrero, J. M. Kremsner, and C. O. Kappe, "Nonthermal microwave effects revisited: On the importance of internal temperature monitoring and agitation in microwave chemistry," *J. Org. Chem.*, 73(1), 36–47, 2008.
- [7] N. R. Baddoo. Stainless steel in construction: A review of research, applications, challenges and opportunities. J. Constr. Steel Res., 64(11) 1199–1206, 2008.
- [8] G. Herting, I. Odnevall Wallinder, and C. Leygraf. Metal release from various grades of stainless steel exposed to synthetic body fluids. *Corros. Sci.*,49(1) 103–111, 2007.
- [9] Parr Vessels, "Safety in the Operation of Laboratory Reactors and Pressure Vessels," no. 230 M, 2016.