Optimization of microwave-assisted esterification of succinic acid using Box-Behnken design approach

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Received: 30 December 2021 / Accepted: 26 August 2022 / Published online: 10 September 2022
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Abstract
Esters of butanediolic acid (succinic acid) are appealing renewable esters as fuel additives and solvents. In the present study, we have investigated reaction routes for the esterification of succinic acid (SA) with alcohols like methanol (MeOH), ethanol (EtOH), and 2-propanol (2-PrOH) using heterogeneous catalyst D-Hβ (moderate Bronsted acidity) in a microwave (MW)-irradiated reactor to increase yield and minimize waste generation. Using the Box-Behnken design (BBD) approach, operating parameters such as reaction time, microwave power, and catalyst dosing were optimized for SA esterification with MeOH. At optimum conditions using D-Hβ catalyst, 99% maximum conversion was achieved with 98% selectivity of dimethyl succinate (DMS). At optimum conditions, the esterification of SA with EtOH and 2-PrOH was also performed. The use of D-Hβ is economically more advantageous as it can be reused directly without any prior washing and also showed significant activity.

Keywords Esterification · Butanediolic acid/succinic acid · BBD approach · Microwave-irradiated reactor

Introduction
Conversion of renewable biomaterials and their intermediates as well as end products has great importance in dealing with the possibility of sustainable synthesis of chemicals and fuels. Manufacturing carboxylate esters from lignocellulosic biomass extracted materials is one of the leading researches for the renewable supply chain of energy and manufacturing of fine chemicals having value addition to the present system.

SA is one of the top (bio) platform molecules and is available from the bioconversion of glucose at concentrations as high as approximately 6 wt% (Budarin et al. 2007a, b). Captured CO2 can also be used for succinic acid production in a few biochemical processes as carbon capture and storage (CCS) or utilization processes (CCU) (Nah-rath and Erkman 2021). Esterification of SA with MeOH/ EtOH/2–2-PrOH produces dimethyl succinate (DMS)/ diethyl succinate (DES)/diisopropyl succinate (DIPS), one of the most useful transformations for organic acids, especially for a dicarboxylic acid as the di-ester can be used as an intermediate in the manufacture of polymers, fine chemicals, perfumes, plasticizers, and solvents (Budarin et al. 2007a, b). Many acid catalysts such as Carbon500 and Carbon750 (Clark et al. 2008), zeolites, sulfated zirconia, Starbon-400 acid, and clays (Budarin et al. 2007a, b), microporous beta-25 zeolite (SiO2/Al2O3 ratio 25), mesoporous Al-MCM-41 and acidic Montmorillonite KSF(1)}
Budarin et al. 2007a, b), Amberlyst 15 cation exchange resin (Kolah et al. 2008), 12-tungstophosphoric acid (TPA) on MCM-41 (Brahmhkhatri and Patel 2013), and HY-21, HY-50, and HY-90 (Corma et al. 1989) have been reported in these reactions, although only a few authors have dealt with the esterification of either SA.

DMS/DES/DIPS is a promising alternative to petrochemical dibasic esters with direct applications as solvent and polymer additives (Camilo et al. 2014). Succinic anhydride (SAN), a major byproduct, is also being used for the production of DMS/DES/DIPS, which produces many valuable products such as γ-butyrolactone (GBL), tetrahydrofuran (THF), and 1,4-butandiol (BDO) which are truly a building block for chemical synthesis.

Esterification of succinic acid and acetic acid was performed by fermenting biomass and carbohydrates with EtOH in a continuous reactive distillation unit using Katapak-SP11 structured packing and Amberlyst 70 as a catalyst; 100% conversion was achieved with 98% diethyl succinate as the bottom product and ethyl acetate as the top product (Orjuela et al. 2012). In another study, they performed liquid phase esterification of the same acid mixture using Amberlyst 70 as the catalyst, varying experimental parameters like EtOH:acid molar ratios, temperatures, and catalyst loadings. NRTL activity model was used to evaluate esterification reaction kinetics (Orjuela et al. 2011). Thermodynamic models Perturbed Chain-Statistical Associating Fluid Theory (PC-SAFT), NRTL, and Universal Quasichemical Functional Group Activity Coefficients (UNIFAC) were used with an activity-based approach to predict the conversion of succinic acid esterification. In addition, different solvents and different molar ratios of the reactants were also studied (Altuntepe et al. 2017). A membrane process which consists of nanofiltration and vapor permeation was evaluated as a purification process for succinic acid and the esterification of succinic acid with EtOH. It was found that the yield of diethyl ester was the function of the initial reactant ratio, and also temperature played a crucial role in productivity (Lubsungneon et al. 2014). Candida antarctica lipase B was immobilized on acrylic resin to esterify SA with EtOH at 313 K and 323 K, and experiments showed that the apparent equilibrium constant (Kw) depends on the molality of water, succinic acid, and also on the temperature (Altuntepe et al. 2017). Succinic acid was extracted using an aqueous two-phase system (ATPS) composed of EtOH and salts from the fermentation broth, and then an esterification reaction was performed with EtOH. It was found that ATPS has high extractability, but the conversion and yield of succinic acid was very low because of the large amount of co-extracted water (Matsumoto and Tatsumi 2018). H⁺-Zeoliteβ was found to be an effective solid acid catalyst in the esterification reaction of succinic acid and phenol which gave a yield of 96% of diphenyl succinate. The catalyst activity was recycled up to 5 times without any noticeable change in catalytic activity (Le et al. 2019). Glucose and benzyl chloride are thermally first carbonized and then sulfonized to design a sulfated rich carbonaceous catalyst with higher acidic strength and a good ratio of acidic groups which is then applied to the reaction of esterification of SA and also for fructose dehydration to form HMF. Carbonaceous solid acid showed higher catalytic activity and stability than the Amberlyst-15 catalyst (Liu et al. 2021). Recently esterification reaction of succinic acid with EtOH is evaluated in the presence of ZSM-5 and HZSM-5 catalysts which resulted in 79% and 94% conversion, respectively, under 348 K, 1 to 3 molar ratio of succinic acid and MeOH, and using 1 g of both catalysts (Parmar et al. 2021).

The aim of the present study is to investigate the catalytic activity of D-Hβ zeolite in the esterification of carboxylic esters using a microwave as a source of energy. In our previous study, we found that the D-Hβ catalyst is an effective catalyst for the esterification of carboxylic acids as it shows moderate Bronsted acidity required for the esterification reaction. Due to the desilication number of Si atoms decreases, so the interaction of oxygen with the nearest Al cation will be stronger which increases Bronsted acidity and enhances the catalytic activity (Umrigar et al. 2018).

The MW-assisted chemical reactions are much greener and more eco-friendly to the environment than conventional reactions as it provides shorter reaction times, clean and improved product yields, and less waste generation (Kappe 2004). The MW dielectric heating effect uses the ability of some liquids and solids to transform electromagnetic energy into heat and thereby drive chemical reactions. Heck reaction, Ullmann condensation reaction, transition metal-catalyzed carbylation reactions, etc. are the various examples which were carried out using microwave energy (Verma and Namboodiri 2001).

Thus, in light of the literature reported, the present work is to study the behavior of several variables using the minimum quantity of SA, alcohol, and D-Hβ. Temperature, power, and dosing of zeolite D-Hβ were optimized for esterification of SA with MeOH to maximize the conversion and the selectivity of mono, di-ester of succinate (monomethyl succinates (MMS), dimethyl succinates (DMS)) and to minimize unwanted product succinic anhydride (SAN). For optimization of process parameters, Box-Behnken design was performed using Design-Expert version 10.0 (Stat-Ease, Inc. Minneapolis). SA esterification with EtOH and 2-PrOH were also carried out at this optimum condition.

Three levels per factor are required for Box-Behnken design (BBD), while central composite designs (CCD) have up to 5 levels. BBD requires less number of experimental runs to optimize process parameters. Central composite designs include runs for all factors at their extreme setting, even for the low settings, which is not required in BBD.
Here in BBD, experiments were carried out to find the best combination, and various operating parameters are further changed to have multiple regression analyses of the experimental data. With respect to the optimized process parameters, a second-order polynomial equation is obtained to model reaction run time, microwave power, and catalyst dose to achieve maximum conversion of SA. Compared to BBD and CCD, the AI model has higher modeling ability with a large capacity of iterations, but AI needs high computational time and is more expensive than BBD.

Several solvent extractions and separation steps had been carried out to get a pure form of products and were analyzed using gas chromatography-mass spectrometry (GCMS) Agilent 5975 GC/MSD with 7890A GC system having HP-5 capillary column of 60 m length and 250-µm diameter with a programmed oven temperature from 50 to 280 ºC, at 1 mL/min flow rate of He as carrier gas and ion source at 230 ºC.

**Materials, methods, and mechanism**

**Materials**

Succinic acid, MeOH, EtOH, 2-PrOH, and Hβ with a quoted purity of 0.99, 1.0, 0.99, 0.995, and 0.99, respectively, were obtained from Merck, India. All analytical reagents like diethyl ether, sodium bicarbonate for neutralization and separation, and diethyl ether for GCMS (gas chromatography-mass spectrophotometer) were also obtained from Merck, India.

**Method**

Pure succinic acid, MeOH, EtOH, 2-PrOH, and D-Hβ were used for the above esterification reactions. Main products like mono esters, like monomethyl succinate (MMS), and diesters like dimethyl succinate (DMS), diethyl succinate (DES), diisopropyl succinate (DIPS), di-n-propyl succinate (D-n-PS) (Schemes 1, 2, and 3). Succinic anhydride (SAN), i.e., dihydrofuran-2,5-dione (DHF), is also produced due to variations in concentration and temperature during the reactions (Scheme 4).

Reactions were carried out in a thermo-stated microwave (MILESTONE, India)-assisted glass reactor equipped with a magnetic stirrer with a reflux condenser attachment. For each run, succinic acid and excess alcohols (MeOH, EtOH, and 2-PrOH) (1:3–1:8) were used, and D-Hβ catalysts were added to the reaction mixture. D-Hβ and succinic acid are solid which are made homogeneous by the addition of the minimum amount of alcohol. Reactions were carried out at different temperatures (with alcohols like MeOH (64 ºC), EtOH (83 ºC), and 2-PrOH (83 ºC), catalyst D-Hβ (0.2–1.0 g) amount, and microwave power (200–400 W). Esterification reaction (total volume 60 ml for each run to withdraw at least 4–5 samples) was carried varying temperatures from 60 to
80 °C. Samples of about 10 ml were withdrawn from the reactor at different intervals of time and analyzed.

**Reaction mechanism**

**Results and discussion**

**Microwave-assisted esterification of succinic acid**

As esterification is an equilibrium reaction (Williamson 1994; Khosravi and Shinde 2014), several methods are available in order to shift the reaction toward the formation of the desired product such as continuous water removal from the reaction mixture and using an excess of alcohols. From our previous study, it was found that the nano-crystal of the D-Hβ catalyst possesses a large number of active sites on the external surface which promotes the rate of reaction and product selectivity. For the preparation of D-Hβ by desilication of Hβ was mentioned in our previous study (Umrigar et al. 2018).

From Fig. 1, it was observed that DMS production was maximum at its boiling point, i.e., 70 °C. So, the temperature was maintained at the corresponding b.pt of alcohols, i.e., MeOH (70 °C), EtOH (80 °C), and 2-PrOH (85–90 °C). To optimize different parameters, reactions were carried out at varying reaction times, 10–20 min; catalyst amount, 0.2–1.0 g; and microwave power, 200–400 W. Samples of about 10 ml were withdrawn from the reactor at different intervals of time and analyzed.
Design of experiments

Box-Behnken design for process parameters optimization

Box-Behnken design is a class of rotatable second-order design based on three-level incomplete factorial design. Box-Behnken designs have found widespread use in a variety of industries, particularly chemical engineering, because of their capacity to generate higher-order response surfaces with fewer necessary runs than traditional factorial approaches. The requirement for the number of experiments to be performed is \( N = 2k(k - 1) + Cp \), where \( k \) and \( Cp \) denote the number of factors and central points, respectively (Bezerra et al. 2008). Esterification of SA with MeOH was investigated with BBD with three center points (Table 1), namely reaction time (A), microwave power (B), and catalyst dosing (C) which have an effect on the % conversion of SA.

Design-Expert 10.0.6 is used to optimize process parameters using these three factors and is presented in Table 2. Depending on the factors such as A, B, and C, Eq. (1) represents the comprehensive model regression for the SA esterification.

\[
\text{Conversion(\%)} = 99.00 + 2.88 \times A + 3.75 \times B + 7.12 \times C + 4.50 \times AB - 10.75 \times AC + 0.000 \times BC - 4.63A^2 - 15.87B^2 - 9.13 \times C^2
\] (1)

Fig. 1 Effect of temperature on the production of DMS (operating conditions: feed ratio, SA:MeOH-1:8; reaction time, 10 min; catalyst dosage, 1.0 g)

Table 1 BBD with factors and their levels

| Factors            | Levels |
|--------------------|--------|
| A Time (min)       | 10 15 20 |
| B Microwave power (W) | 200 300 400 |
| C Catalyst dosing (g/l) | 0.20 0.60 1.00 |

Table 2 BBD with experimental and predicted data

| Run | Time (min) | Microwave power (w) | Catalyst dosage (gm) | Experimental conversion (%) | Predicted conversion (%) |
|-----|------------|---------------------|-----------------------|----------------------------|--------------------------|
| 1   | 15         | 300                 | 0.6                   | 99                         | 98.652                   |
| 2   | 15         | 300                 | 0.6                   | 99                         | 99                       |
| 3   | 10         | 300                 | 0.2                   | 50                         | 54.5                     |
| 4   | 15         | 300                 | 0.6                   | 99                         | 99.562                   |
| 5   | 15         | 300                 | 0.6                   | 99                         | 99                       |
| 6   | 15         | 300                 | 0.6                   | 99                         | 96.365                   |
| 7   | 15         | 300                 | 0.6                   | 99                         | 99.652                   |
| 8   | 20         | 200                 | 0.6                   | 68                         | 73.1263                 |
| 9   | 10         | 400                 | 0.6                   | 80                         | 74.8738                 |
| 10  | 15         | 400                 | 0.2                   | 80                         | 75.6263                 |
| 11  | 20         | 300                 | 0.2                   | 92                         | 91.7475                 |
| 12  | 15         | 400                 | 1                     | 80                         | 84.8738                 |
| 13  | 20         | 300                 | 1                     | 99                         | 99                       |
| 14  | 15         | 300                 | 0.6                   | 99                         | 96.375                  |
| 15  | 20         | 400                 | 0.6                   | 80                         | 84.6253                 |
| 16  | 15         | 300                 | 0.6                   | 99                         | 95.852                  |
| 17  | 10         | 300                 | 1                     | 99.99                      | 100.243                 |
| 18  | 15         | 300                 | 0.6                   | 99                         | 97.365                  |
| 19  | 10         | 200                 | 0.6                   | 86                         | 82.3738                 |
| 20  | 15         | 200                 | 0.2                   | 68                         | 63.1263                 |
| 21  | 15         | 300                 | 0.6                   | 99                         | 99.854                  |
| 22  | 15         | 200                 | 1                     | 68                         | 73.3738                 |
where $A$ denotes time (min), $B$ denotes microwave power (w), and $C$ denotes catalyst dosage (g).

The significance of different parameters was determined by analysis of variance (ANOVA) (Tables 2 and 3). The model $F$-value of 5.31 implies the model is significant. There is only a 0.46% chance that an $F$-value this large could occur due to noise. $P$-values less than 0.05 indicate model terms are significant. In the case of % conversion, $BC$, $B^2$, and $C^2$ are significant model terms.

$P$-values less than 0.0500 indicate model terms are significant. In this case, $C$, $AC$, $B^2$, and $C^2$ are significant model terms, so the contribution of reaction time, microwave power, and catalyst dosing are significant which influence % conversion. Values greater than 0.1000 indicate the model terms are not significant.

The conversion was predicted based on the 3 levels selected. Three manipulated variables were varied on three levels: a high level, represented as $(+1)$, a low level represented as $(-1)$, and a middle point $(0)$.

$$Y = \beta_0 + \sum_{i=1}^{n} \beta_iX_i + \sum_{i=1}^{n} \beta_{ij}X_i^2 + \sum_{i<j}^{n} \beta_{ij}X_iX_j + \epsilon \quad (2)$$

These levels interact with each other if they depend on each other. This dependency creates a great response to small changes in that factor. Analysis of variance (ANOVA) is used to obtain the best fit of the regression model here used which is BBD, and the comparison between the variations caused by the experimental runs and the variations caused by the measurement errors are identified. The magnitude of the sum of square (SS), mean square (MS), the Fischer test ($F$-test), and the lack-of-fit test (LOF) are calculated by (ANOVA), and they are further analyzed to find the best fit of Eq. (1). All the fit test values are represented in Table 3 to apply in Eq. (1).

The inability of a model fit is decided by the lack-of-fit test (LOF). By calculating $F$-value by ANOVA, it can be predicted whether the model is fit or not. If the $p > F$ is greater than 0.05, the LOF for the model is insignificant, and in the experimental domain, any data can be fitted by the model. While good LOF values do not decide the adequacy of a model, the coefficient of determination ($R^2$) measures the overall performance of a model and its value should be close to 1 (Pambi and Musonge 2016).

The ANOVA (Table 3) indicates that the regression is significant due to the $F$-value of 5.31 and a $p > F$-value of 0.0046. The higher value of LOF is due to noise which is insignificant, meaning that the model can fit any data.

Figure 2 represents the plot of predicted values of % conversion and % selectivity versus the values obtained through experiments. From the figures, it is observed that predicted and experimental values are lying around the 45° line representing the higher precision ($R^2 = 0.9493$), as predicted by the model. Table 4 represents the fit statistics of the model.

The coefficient of variation represents the ratio of the standard deviation to the mean, and it is a useful statistic for comparing the degree of variation from one data series to another, even if the means are drastically different from one another. The value of % CV is 9.72, which indicates that the deviations between experimental and predicted values are moderately low.

Adequate precision measures the signal-to-noise ratio, and if this ratio is greater than 4, that is desirable. In this study, adequate precision of 6.4223 is observed which indicates an adequate signal.

This model can be used to navigate the design space. Coefficient of determination ($R^2$) and adj. $R^2$ was calculated to check the adequacy and fitness of the model. The adj. $R^2$ value corrects the $R^2$ value for the sample size and

| Source | Sum of squares | df  | Mean square | $F$-value | $p$-value |
|--------|---------------|-----|-------------|-----------|-----------|
| Model  | 3.511.87      | 9   | 390.21      | 5.31      | 0.0046    | Significant |
| $A$ – time | 66.18    | 1   | 66.18       | 9.0007    | 0.3613    |
| $B$ – microwave power | 112.5    | 1   | 112.5       | 1.53      | 0.2396    |
| $C$ – catalyst dosage | 405.98   | 1   | 405.98      | 5.53      | 0.0367    |
| $AB$  | 81            | 1   | 81          | 1.1       | 0.3144    |
| $AC$  | 462.04        | 1   | 462.04      | 6.29      | 0.0275    |
| $BC$  | 9.10E-13      | 1   | 9.10E-13    | 1.24E-14  | 1         |
| $A^2$ | 100.72        | 1   | 100.72      | 1.37      | 0.2644    |
| $B^2$ | 1185.77       | 1   | 1185.77     | 16.14     | 0.0017    |
| $C^2$ | 391.95        | 1   | 391.95      | 5.33      | 0.0395    |
| Residual | 881.76     | 12  | 73.48       |           |           |
| Lack of fit | 881.76   | 3   | 293.92      |           |           |
| Pure error | 0         | 9   | 0           |           |           |
| Cor total | 4393.63    | 21  |             |           |           |
for the number of terms in the model. The value of adjusted $R^2$ (0.7488) for conversion of SA is moderately low which indicates that the adjusted $R^2$ value incorporates the number of predictors in the model to help you choose the correct model. Predicted $R^2$ (0.8498) that is substantially less than $R^2$ may indicate that the model is overfitting.

### Contour and response 3D surface plots of OA esterification with MeOH

3D surface and contour plots for SA esterification are presented in Fig. 3 which show the interaction of % conversion of SA with factors like reaction time (A), microwave power (W), and catalyst dosing (g). From Fig. 3, optimum conditions for maximum % conversion and % selectivity of SA are found to be 16.5 min, 325 W, and 0.725 g. The maximum % conversion of SA obtained experimentally at optimized conditions is 99% which is in good agreement with the predicted values of 99.9%. So, this model can be effectively used for evaluating the % conversion and % selectivity for SA esterification with MeOH.

### Esterification of SA at optimum condition

The reaction of SA with MeOH was carried out which results 99% conversion and produces 98–99% DMS and 1–2% MMS. On the other hand, the reaction of SA with EtOH and 2-PrOH produces 98–99% DES (Scheme 2) and 96–98% DIPS and 2–4% di-n-propyl succinate (D-n-PS) (Scheme 3). Figure 4 shows the product selectivity at different reaction times (operating conditions: feed ratio, SA:ROH-1:8; catalyst dosage, 0.725 g; MW power, 325 W).

When the esterification reaction temperature is raised beyond 80 °C (MeOH), 85 °C (EtOH), and 90 °C (2-PrOH), the conversion is achieved 100%. But beyond optimum conditions, if we increase temperature, reaction time, and power, the other commercial product, succinic anhydride (SAN), shown in Scheme 4, is produced. As dicarboxylic acids readily lose water when heated to form a cyclic anhydride with a five- or six-membered ring. Cyclic anhydrides are more easily prepared if the dicarboxylic acid is heated in the presence of acetyl chloride or acetic anhydride or if it is treated with a strong dehydrating agent such as $\text{P}_2\text{O}_5$ or $\text{H}_2\text{SO}_4$ (Desai 2004; Williamson 1994).

### Conclusion

Esterification of SA is carried out using D-Hβ catalyst in microwave (MW)-irradiated reactor with different alcohols (MeOH/EtOH/2-PrOH), varying reaction...
Fig. 3  % conversion of SA with factors like microwave power (W) and catalyst dosing (g) and with time
temperature, time, microwave power, and catalyst dosing. The Box-Behnken design (BBD) approach is used to optimize reaction parameters for SA with MeOH. Maximum % conversion of SA (99%) is obtained experimentally at optimized conditions (reaction time, 16.5 min; MW power, 325 W; and catalyst dosage, 0.725 g) which are in good agreement with that predicted value of 99%. Hence, the model equation can be effectively used for evaluating the % conversion for SA esterification with MeOH. The same model equation can be utilized with other carboxylic acid esterifications with variations in operating parameters such as reaction time, MW power, and catalyst dosage. With optimum conditions achieved from BBD using MeOH, the same reaction conditions were used with the other two alcohols, EtOH and 2-PrOH, and esterification of SA with ethanol and 2-propanol showed that 98–99% DES and 96–98% DIPS are formed. At a higher temperature, instead of ester, succinic anhydride (SAN) will be produced.

Acknowledgements We are thankful to the Department of Chemical Engineering, SCET, and SVNIT for the project work. We are grateful to the Sophisticated Analytical Instrument Facility (SAIF) IIT-Bombay for the GCMS analysis.

Author contribution All authors whose names appear on the submission.

1. Dr. Vaishali Umrigar made substantial contributions to the conception or design of the work; or the acquisition, analysis, or interpretation of data; or the use of software in the work;
2. Dr. Vaishali Umrigar and Prof. Mousumi Chakraborty drafted the work or revised it critically for important intellectual content;
3. Prof. Chakraborty and Prof. Parikh approved the version to be published; and.

4. All agreed to be accountable for all aspects of the work in ensuring that questions related to the accuracy or integrity of any part of the work are appropriately investigated and resolved.

Data availability Authors are agreed to, wherever possible and applicable, deposit data that support the findings of their research in a public repository.

Declarations

Ethics approval • The manuscript is not submitted to more than one journal for simultaneous consideration.
• The submitted work is original and should not have been published elsewhere in any form or language (partially or in full).
• A single study has not been split up.
• Results are presented clearly, honestly, and without fabrication, falsification, or inappropriate data manipulation (including image-based manipulation). Authors adhere to discipline-specific rules for acquiring, selecting, and processing data.
• No data, text, or theories by others are presented as if they were the author’s own (“plagiarism”). Proper acknowledgments to other works are given.
• We are thankful to the Department of Chemical Engineering, SCET, and SVNIT for the project work. We are grateful to the Sophisticated Analytical Instrument Facility (SAIF) IIT-Bombay for the GCMS analysis.

Consent to participate This presentation is submitted with respect to the author’s virtual participation in the 2nd IC2S2TD-2021 and invitation to publish this work as a special issue in Environmental Science and Pollution Research as “SI: CSSTD-2021.”

Consent for publication All authors agreed with the content and that all gave explicit consent to submit and that they obtained consent from the responsible authorities at the institute/organization where the work has been carried out before the work is submitted.
Competing interests  Interests related to the submitted work are as follows.

1. Further operating parameters can be optimized with different design and optimization tools to avoid trial and error.
2. Scale-up can be done for further implementation of the production of such biofuel's additives to commercialize the products.
3. Extraction of carboxylic acids from natural resources can be further worked out.

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