Optimization of Microwave Assisted Esterification of Succinic Acid Using Box-Behnken Design Approach

Vaishali Rakesh Umrigar (vaishali.umrigar@scet.ac.in)
Sarvajanik College of Engineering and Technology https://orcid.org/0000-0001-6537-700X
Mousumi Chakraborty
Sardar Vallabhbhai National Institute of Technology
Parimal A. Parikh
Sardar Vallabhbhai National Institute of Technology

Research Article

Keywords: Esterification, Butanedioic acid/ Succinic acid, BBD approach, Microwave irradiated reactor

Posted Date: February 2nd, 2022

DOI: https://doi.org/10.21203/rs.3.rs-1206807/v1

Abstract

Esters of Butanedioic acid (succinic acid) are appealing renewable esters as a fuel additive and solvents. In present study, we have investigated reaction routes for the esterification of Succinic acid (SA)with alcohols like MeOH, ethanol and 2-PrOH using heterogeneous catalyst D-Hβ (moderate bronsted acidity) in Microwave (MW) irradiated reactor to increase yield and minimise waste generation.Using Box-Behnken design (BBD) approach, operating parameters such as reaction time, microwave power and catalyst dosing were optimized for SA esterification with methanol. At optimum condition using D-Hβ catalyst, 99% maximum conversion was achieved with 98% selectivity of Dimethyl Succinate (DMS). At optimum condition, esterification of SA with ethanol and propanol were also performed.Use of D-Hβ is economically more advantageous as it can be reused directly without any prior washing and also showed significant activity.

1. Introduction

Conversion of renewable biomaterials and their intermediates as well as end products has great importance to deal with the possibility of sustainable synthesis of chemicals and fuels. Manufacturing of carboxylate esters from lignocellulosic biomass extracted materials is one of the leading researches for 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%. Esterification of SA with methnol/ethanol/2-Propanol 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 diester can be used as an intermediate in the manufacture of polymers, fine chemicals, perfumes, plasticizers and solvents. Many acid catalysts have been reported in these reactions, although only a few authors have dealt with the esterification of either SA (Budarin et al.2007). DMS/DES/DIPS is a promising alternative to petrochemical dibasic esters with direct applications as solvent and polymer additive (Camilo et al.2014). SAN, a major byproduct is also being used for productin of DMS/DES/DIPS, which produces many valuable products such asγ-Butyrolactone (GBL),tetrahydrofuran (THF) and 1,4-Butanediol (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 ethanol in continuous reactive distillation unit using Katapak-SP11 structured packing and Amberlyst 70 as catalyst. 100% conversion was achieved with 98% diethyl succinate as bottom product and ethyl acetate as top product (Orjuela et al.2012). In another study they performed liquid phase esterification of the same acid mixture using Amberlyst 70 as catalyst varying experimental parameters like ethanol:acid molar ratios, temperatures and catalyst loadings. NRTL activity model were used to evaluate esterification reaction kinetics (Orjuela et al.2011). A membrane process which consist of a nanofiltration and vapor permeation was evaluated as purification process for succinic acid and the

esterification of succinic acid with ethanol. It was found that yield of diethyl ester was the function of initial reactant ratio and also temperature played crucial role in productivity (Lubsungneon et al. 2014). Candida antarctica lipase B was immobilized on acrylic resin to esterify SA with ethanol at 313 and 323 K and experiments showed that Apparent equilibrium constant (K_m) depends on molality of water, succinic acid and also on the temperature (Altuntepe et al. 2017). Succinic acid was extracted using aqueous two-phase system (ATPS) composed of ethanol and salts, from fermentation broth and then esterification reaction was performed with ethanol. It was found that ATPS having high extractability but conversion and yield of succinic acid was very low because of large amount of co-extracted water (Matsumoto and Tatsumi 2018). H^+-Zeoliteβ was found to be effective solid acid catalyst in the esterification reaction of succinic acid and phenol which gave yield of 96% of diphenyl succinate. Catalyst activity was recycled upto 5 times without any noticeble change in catalytic activity (Le et al. 2019). Glucose and Benzyl chloride are thermally first carbonized and then sulfonized to design a sulphated rich carbonaceous catalyst with higher acidic strength and good ratio of acidic groups which 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 Amberlyst-15 catalyst (Liu et al.2021). Recently esterification reaction of succinic acid with ethanol is evaluated in presence of ZSM-5 and HZSM-5 catalyst which resulted 79% and 94% conversion respectively under 348 K, 1 to 3 molar ratio of succinic acid and methanol and using1g of both catalyst (Parmar et al.2021).

The aim of the present study is to investigate catalytic activity of D-Hβ zeolite in the esterification of carboxylic esters using microwave as a source of energy. In our previous study we found that D-Hβ catalyst is an effective catalyst for estertrification of carboxylic acids as it shows moderate Bronsted acidity required for esertification reaction. Due to desilication number of Si atom decreases, so 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 microwave (MW) assisted chemical reactions are much more greener and ecofriendly 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, and transition metal catalyzed carbonylation reactions etc are the various examples which were carried 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 minimum quantity of SA, alcohol and D-Hβ. Temperature, power, dosing of zeolite D-Hβ were optimized for esterification of SA with methanol to maximize the conversion and the selectivity of mono, di-ester of succinate (Monomehtyl Succinates (MMS), Dimethyl Succinates (DMS)) and to minimise unwanted product Succinic anhydride (SAN). For optimization of process parameters Box-Behnken design was performed using Design-Expert Version10.0 (Stat-Ease, Inc. Minneapolis). SA esterification with ethanol and 2-propanol were also carried out at this optimum condition.

Several solvent extraction and separation steps had been carried out to get 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 micrometer diameter with a programmed oven temperature from 50 to 280°C, at 1 mL/ min flow rate of He as carrier gas and ion source at 230°C.

1. **2.Materials, Methods and mechanism**:
2. **2.1Materials**: Succinic Acid, Methanol, Ethanol, 2-Propanol 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 diethylether, sodium bicarbonate for neutralization and separation and diethylether for GCMS (Gas chromatography Mass Spectrophotometer) were also obtained from Merck, India.
3. **2.2 Method**:Pure Succinic Acid, Methanol, Ethanol, 2-Propanol 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) (Scheme 1, 2 &3). Succinaic Anhydrides (SAN)i.e. Dihydrofuran-2,5-dione (DHF) is also produced due to variation 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 alcohols (excess) were used and D-Hβ catalysts were mixed to prepare reaction mixture. Reaction was carried out at different temperatures, catalyst (D-Hβ) amount and microwave power. Esterification reaction (total volume 60 ml for each run) was taken and temperature was varied from 60-80ºC. Samples of about 10ml were withdrawn from the reactor at different intervals of time and analyzed.

2.3.Reaction-Mechanism:

2.4 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 towards formation of the desired product such as continuous water removal from the reaction mixture and using excess of alcohols. From our previous study it was found that nano-crystal of D-Hβ catalyst possesses large number of active sites on the external surface which promotes rate of reaction and product selectivity. For 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. 70ºC. So temperature was maintained at corresponding b.pt of alcohols i.e. MeOH (70ºC), Ethanol (80 ºC) and 2-PrOH (85-90ºC).To optimize different parameters, reactions were carried out varying reaction time (10-20 min), catalyst amount (0.2-1.0 g) and microwave power (200-400W). Samples of about 10ml were withdrawn from the reactor at different intervals of time and analyzed.

Fig:1 Effect of temperature on the production of DMS

2. Materials, Methods And Mechanism

2.1Materials: Succinic Acid, Methanol, Ethanol, 2-Propanol 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 diethylether, sodium bicarbonate for neutralization and separation and diethylether for GCMS (Gas chromatography Mass Spectrophotometer) were also obtained from Merck, India.

2.2 Method: Pure Succinic Acid, Methanol, Ethanol, 2-Propanol 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) (Scheme 1, 2 &3). Succinaic Anhydrides (SAN)i.e. Dihydrofuran-2,5-dione (DHF) is also produced due to variation 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 alcohols (excess) were used and D-Hβ catalysts were mixed to prepare reaction mixture. Reaction was carried out at different temperatures, catalyst (D-Hβ) amount and microwave power. Esterification reaction (total volume 60 ml for each run) was taken and temperature was varied from 60-80ºC. Samples of about 10ml were withdrawn from the reactor at different intervals of time and analyzed.

2.3.Reaction-Mechanism:

2.4 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 towards formation of the desired product such as continuous water removal from the reaction mixture and using excess of alcohols. From our previous study it was found that nano-crystal of D-Hβ catalyst possesses large number of active sites on the external surface which promotes rate of reaction and product selectivity. For 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. 70ºC. So temperature was maintained at corresponding b.pt of alcohols i.e. MeOH (70ºC), Ethanol (80 ºC) and 2-PrOH (85-90ºC).To optimize different parameters, reactions were carried out varying reaction time (10-20 min), catalyst amount (0.2-1.0 g) and microwave power (200-400W). Samples of about 10ml were withdrawn from the reactor at different intervals of time and analyzed.

3. Results And Discussion

3.1. Design of experiments

3.1.1. Box-Behnken design (BBD) for process parameters optimization

Box benkhen design is a class of rotable 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 of number of experiments to be performed, N=2k(k-1) + Cp where k and Cp denotes the number of factors and central points respectively (Bezerra et al.2008). Esterification of SA with methanol were investigated with BBD with three centre points (Table 1), namely Reaction time (A), Microwave Power (B) and Catalyst dosing(C) which have effect on % conversion of SA.

Table 1
BBD with factors and their levels

	Factors	Levels		
		1	2	3
A	Time(min)	10	15	20
B	Microwave Power (W)	200	300	400
C	Catalyst dosing (g/l)	0.20	0.60	1.00

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) representsthe comprehensive model regression for the SA esterification.

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

Where A=Time (min) B=Microwave Power (w) C=Catalyst Dosage (g)

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

Table.3 Response 1: Conversion

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3511.87	9	390.21	5.31	0.0046	significant
A-Time	66.18	1	66.18	0.9007	0.3613	
B-Microwave Power	112.50	1	112.50	1.53	0.2396	
C-Catalyst Dosage	405.98	1	405.98	5.53	0.0367	
AB	81.00	1	81.00	1.10	0.3144	
AC	462.04	1	462.04	6.29	0.0275	
BC	9.095E-13	1	9.095E-13	1.238E-14	1.0000	
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	0.0000	9	0.0000			Not significant
Cor Total	4393.63	21				

Significance of different parameters was determined by Analysis of variance (ANOVA) (Table 2 & 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 case of % conversion BC, B^2, C^2 are significant model terms.

P-values less than 0.0500 indicate model terms are significant. In this case C, AC, B^2, C^2 are significant model terms, so contribution of Reaction time, Microwave Power and Catalyst dosing are significant which influences on % conversion. Values greater than 0.1000 indicate the model terms are not significant.

Figure 2 Plot of predicted values versus experimental values of % conversion for SA acid esterification with methanol

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

Table 4
Fit Statistics

Std. Dev.	**8.57**	**R^2**	**0.9493**
Mean	88.23	**Adjusted R^2**	0.7488
C.V. %	9.72	**Predicted R^2**	0.8498
		Adeq Precision	6.4223

Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. Here ratio of 6.422 indicates an adequate signal. This model can be used to navigate the design space.

3.1.2. Contour and Response 3D surface plots of OA esterification with methanol

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, 325W and 0.725g. Maximum % conversion of SA obtained experimentally at optimized conditions is 99% which is in good agreement with that predicted values 99.9%. So, this model can be effectively used for evaluating the % conversion and % selectivity for SA esterification with methanol.

3.2 Esterification of SA at optimum condition

Reaction of SA with methanol were carried out which results 99% conversion and produces 98-99% DMS and 1-2% MMS. On the other hand reaction of SA with Ethanol and 2-propanol produces 98-99% DES (Scheme.2) and 96-98% DIPS and 2-4% n-DPS (Scheme 3). Fig. 4 shows the product selectivity at diiferent reaction time.

When estererification reaction temperature raised beyond 80°C (Methanol), 85°C (Ethanol) and 90°C (Propanol), the conversion is achieved 100%. But beyond optimum condition 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 a 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 P_2O_5 or H_2SO_4 (Desai 2004,Williamson 1994).

4. 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 temperature, time, microwave power, catalyst dosing. Box-Behnken design approach is used to optimize reaction parameters for SA with MeOH. Maximum % conversion of SA obtained experimentally at optimized conditions is 99% which is in good agreement with that predicted values 99.9%. Hence the model equation can be effectively used for evaluating the %

conversion for SA esterification with methanol. At optimum condition, esterification of SA with ethanol and 2-propanol were also performed. which 98-99% DES and 96-98% DIPS respectively. It is also observed that at higher temeprature and microwave power SAN is mainly produced.

Abbreviations

MW- Microwave

SA- Succinic Acid

DMS- Dimethyl succinate

DES-Diethyl Succinate

n-DPS-Di-n-Propyl Succinate

DIPS- Diisopropyl Succinate

MWI- Microwave Irradiation (MWI)

GCMS-Gas chromatography-Mass Spectroscopy

SAN- Succinic Anhydride

Declarations

Ethical 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 is 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 acknowledgements 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 Sophisticated Analytical Instrument facility (SAIF) IIT-Bombay for the GCMS analysis.

Consent to Participate: This presentation is submitted with respect to the author virtual participation in 2nd IC^{2}S^{2}TD-2021 and invitation to publish this work as a special issue in Environmental Science and

Pollution Research as “SI: CSSTD-2021”.

Consent to Publish: 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.

Authors Contributions: All authors whose names appear on the submission

1) 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) Drafted the work or revised it critically for important intellectual content;

3) Approved the version to be published; and

4) Agree 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.

Funding: The authors declare that no funds, grants, or other support were received during the preparation of this manuscript.

Competing Interests: Interests related to the submitted work as follows:

1. Further operating parameters can be optimized with different design and optimization tools to avoid trial & 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.

Availability of data and materials:

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

Acknowledgments

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

Conflicts of Interest

No conflict of interest.

References

1. Altuntepe E, Greinert T, Hartmann F et al (2017) Thermodynamics of enzyme-catalyzed esterifications: I. Succinic acid esterification with ethanol. Appl. Microbiol. Biotechnol. 101:5973-5984. https://link.springer.com/article/10.1007%2Fs00253-017-8481-4
2. Bezerra MA, Santelli RE, Oliveira EP et al (2008) Review Response surface methodology (RSM) as a tool for optimization in analytical chemistry. Talanta 76:965–977. https://doi.org/10.1016/j.talanta.2008.05.019
3. Budarin V, Luque R, Macquarrie DJ et al (2007) Towards a Bio-Based Industry: Benign Catalytic Esterifications of Succinic Acid in the Presence of Water, Chem. Eur J 13:6914–6919. https://doi.org/10.1002/chem.200700037
4. Camilo SL, Luuk AM, Wielen V et al (2014) Green upgrading of succinate using dimethyl carbonate for a better integration with fermentative production. Chem Eng J 235:52–60. .https://doi.org/10.1016/j.cej.2013.09.017
5. Desai KR (2004) Mechanism of microwave dielectric heating. Green chemistry- microwave synthesis. 1:12
6. Kappe CO (2004) Controlled microwave heating in Modern organic synthesis.Angew. Chem Int Ed 43:6250–6284. https://doi.org/10.1002/anie.200400655
7. Khosravi M, Shinde (2014) S.N.Kinetic study of esterification of adipic acid with methanol over Amberlyst 15.Int.J. ChemTech Res 6:4872–4877. .https://sphinxsai.com/2014/ch_vol6_no11/5/(4872-4877)%20014.pdf
8. Le S, Nishimura S, Ebitani K (2019) Direct esterification of succinic acid with phenol using zeolite beta catalyst. Catal Commun 122:20–23. https://doi.org/10.1016/j.catcom.2019.01.006
9. Liu H, Peng Q, Ren J et al (2021) Synthesis of a sulfatedgroupriched carbonaceous catalyst and its application in the esterifcation of succinic acid and fructose dehydration to form HMF. J Iran Chem Soc 18:2649–2656. https://doi.org/10.1007/s13738-021-02220-0.)
10. Lubsungneon J, Srisuno S, Rodtong S et al (2014) Nanofiltration Coupled with Vapor Permeation-Assisted Esterification as an Effective Purification Step for Fermentation-Derived Succinic Acid,J. Membr. Sci 459:132–142. https://doi.org/10.1016/j.memsci.2014.02.006
11. Matsumoto M, Tatsumi M (2018) Extraction and Esterification of Succinic Acid Using Aqueous Two-phase Systems Composed of Ethanol and Salts. 25:101–107. https://doi.org/10.15261/serdj.25.101
12. Orjuela A, Kolah A, Lira CT et al (2011) Mixed Succinic Acid / Acetic Acid Esterification with Ethanol by Reactive Distillation Ind. Eng Chem Res 50:9209–9220. https://doi.org/10.1021/ie200133w
13. Orjuela A, Yanez AJ, Santhanakrishnan A et al (2012) Kinetics of Mixed Succinic Acid/Acetic Acid Esterification with Amberlyst 70 Ion Exchange Resin as Catalyst. Chem Eng J 188:98–107. .https://doi.org/10.1016/j.cej.2012.01.10.3
14. Parmar JA, Gandhi DR, Chopda LV et al (2021) Esterification of bioplatform molecule succinic acid using ZSM-5 and HZSM-5 catalysts 63. 75–83. https://doi.org/10.1080/00194506.2019.1699872

15. Umrigar V, Chakraborty M, Parikh PA (2018) Catalytic activity of zeolite Hβ for the preparation of fuels' additives: Its product distribution and scale up calculation for the biofuel formation in a microwave assisted batch reactor. J Environ Chem Eng 6:6816–6827. https://doi.org/10.1016/j.jece.2018.10.059
16. Verma RS, Namboodiri VV (2001) Solvent-free preparation of ionic liquids using a household microwave oven. Pure Appl Chem 73:1309–1313. .https://doi.org/10.1351/pac200173081309
17. Williamson KL (1994) Synthesis of esters by Fisher Esterification, Macroscale and Microscale Organic Experiments, 2nd Ed., Houghton Mifflin, Boston,385

Scheme

Scheme 1 to 4 is available in supplementary section.

Figures

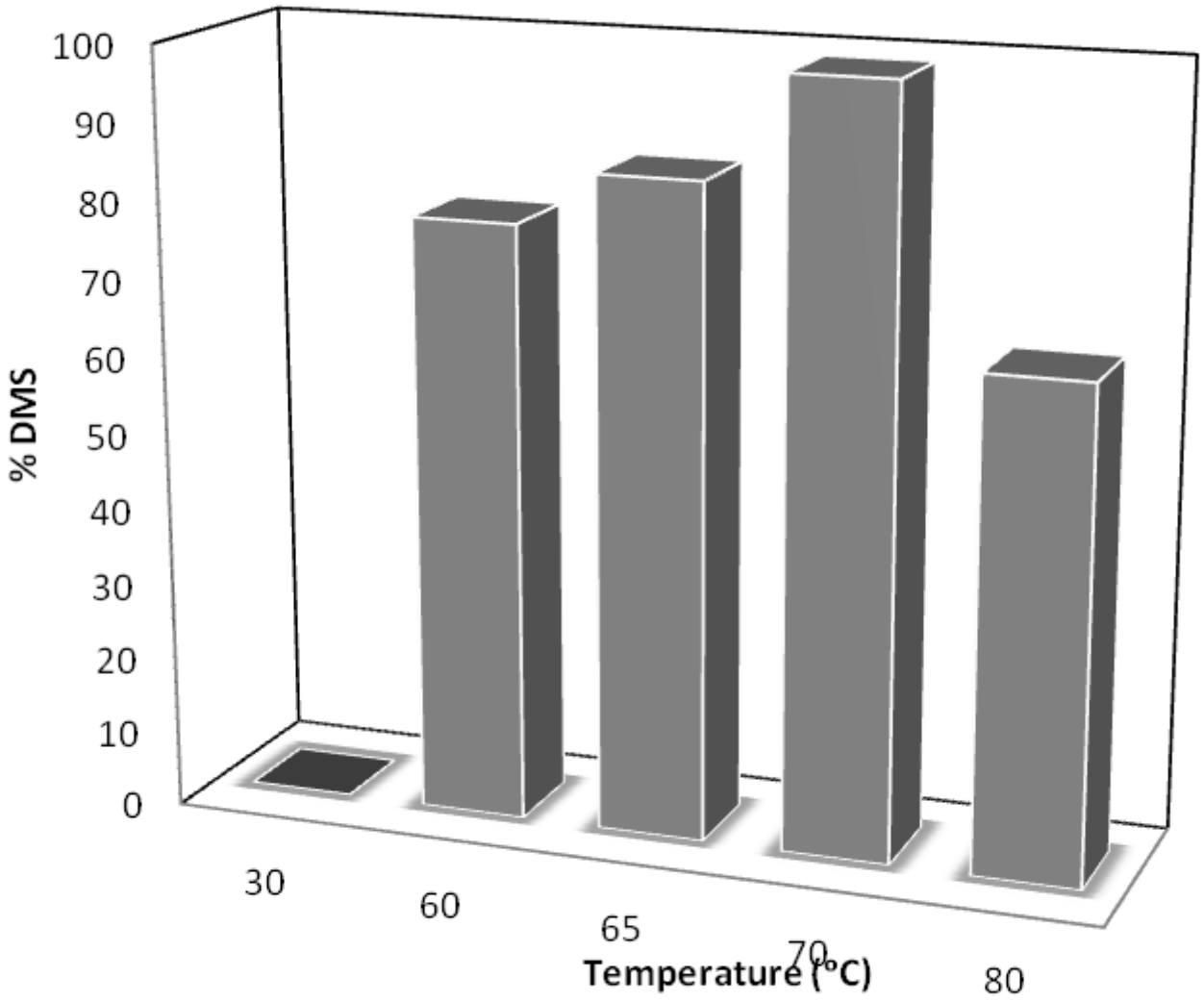

Figure 1

Effect of temperature on the production of DMS

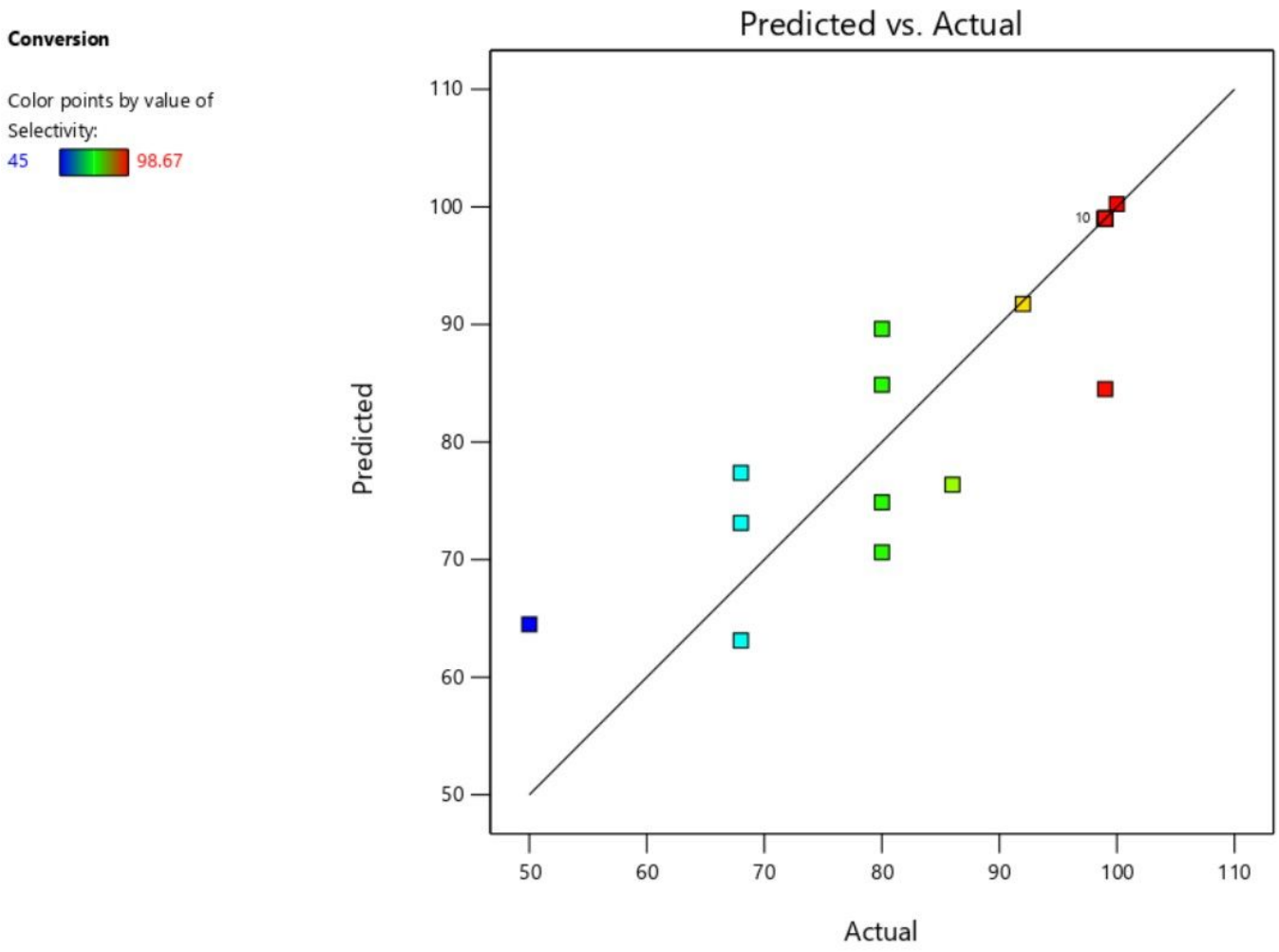

Figure 2

Plot of predicted values versus experimental values of % conversion for SA acid esterification with methanol

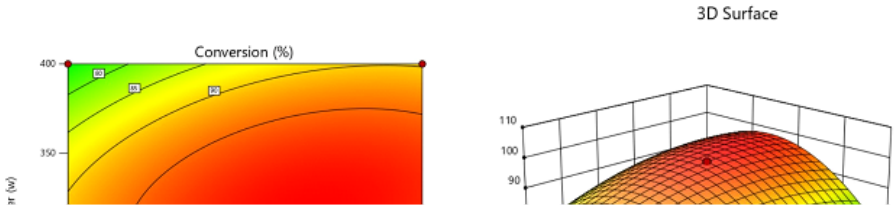

Figure 3

3 % conversion of SA with factorslike Microwave Power (W) and Catalyst dosing(g) and with time.

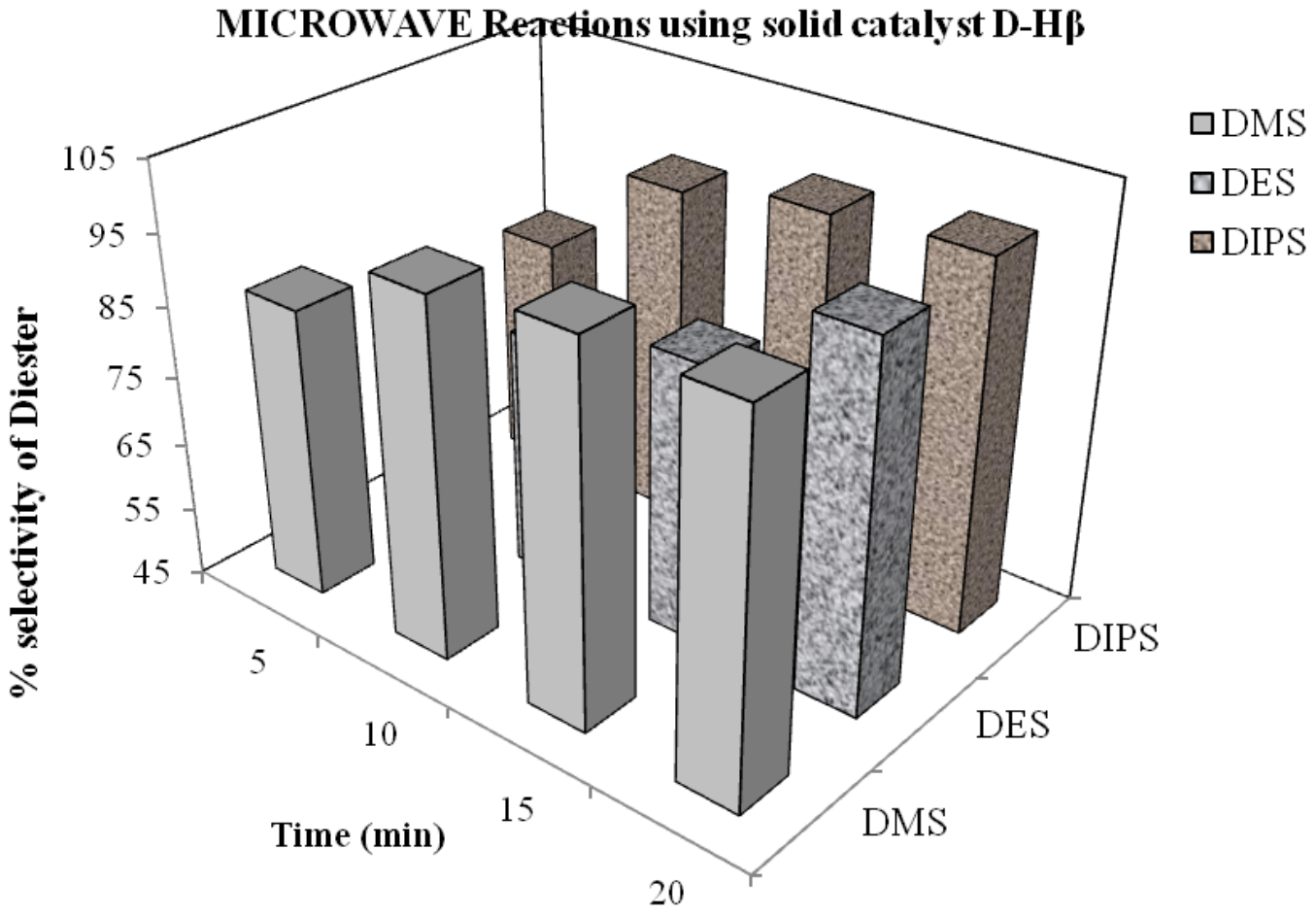

Figure 4

Esterification of SA with (a) % selectivity of Diesters

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- Onlinefloatimage1.png
- Onlinefloatimage2.png
- Onlinefloatimage3.png
- Scheme04.png