One-pot synthesis of dimethyl succinate from D-fructose using Amberlyst-70 catalyst

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ABSTRACT

Dimethyl succinate (DMS), an important building block of bio-based platform chemicals, was produced from D-fructose under one-pot and metal-free conditions for the first time. In the presence of 1.5 mmol D-fructose, 75 mg Amberlyst-70 and 10 bar O_2, 31% of DMS along with 23% of methyl levulinate (ML) was yielded at 130°C for 4 h. The effects of different reaction parameters as well as side products from the solvent on the reaction efficiency were systematically investigated. In order to portray the scope of the process, various biomass derivatives, such as 5-hydroxymethylfurfural, furfural, furfuryl alcohol, etc., were used as promising starting materials for the one-pot synthesis of DMS using the same catalytic system. Besides, the stability of the catalyst was assessed, and the reaction pathway was also proposed based on the experimental results and observations.

1. Introduction

In the context of green and sustainable chemistry, the valorization of renewable biomass resources into high value-added commodity and chemicals has been attracting much attention. Succinic acid (SA), which has been identified as one of the top 10 prospective building blocks from biomass, is used as an important intermediate for the production of various chemicals and polymers, such as 1,4-butanediol, γ-butyrolactone, tetrahydrofuran, pyrrolidone, succinates, poly(butylene succinate) (PBS), etc. [1–4]. Dimethyl succinate (DMS) is one of the important succinates that finds versatile applications. For example, DMS has been widely used as a food additives, as well as in the manufacture of fragrances, pharmaceuticals, agrochemicals, and other fine chemicals (Fig. 1) [3,5,6]. It is one of the solvents used in the commercial dibasic esters mixture [7]. Therefore, research on the synthesis of DMS is always a hot topic in both academia and industry.

Generally, DMS is produced *via* the methyl-esterification of SA using sulfuric acid as the catalyst. Len's group reported the quantitative chemical conversion of SA to DMS at 65°C in the presence of H_2SO_4 under a microwave-assisted continuous flow reaction regime [8]. Dawar et al. yielded 96% of DMS from SA and tert-butyl methyl ether using H_2SO_4 as the catalyst under reflux conditions [9]. In addition to these

promising results, much efforts has been devoted toward this reaction using heterogeneous catalysts to overcome the drawbacks of homogeneous catalysts, such as system corrosion, catalyst recycling, product separation etc. Fabian et al. supported methanesulfonic acid on alumina, and obtained 97% yield of DMS at 80 °C under microwave irradiation for 8 min [10]. Zhang et al. developed a poly ionic liquid-based catalyst, which also showed the excellent esterification of SA to DMS (~100% yield) under 85 °C [11]. More recently, Jumanah et al. used halloysite, a natural clay with a hollow tubular structure, for the esterification of SA, and reported 96% yield for DMS at 170°C for 24 h [12].

On the other hand, new routes have been explored for the synthesis of DMS from different starting materials, such as succinic acid anhydride [5], *cis*-butenedioic anhydride [13], 1,4-butanediol [14], and levulinic acid/ester (LA/E) [3,6,15,16] (Fig. 1). Among them, the Beayer-Villiger (B-V) oxidation of LA/E (which can be readily produced from lignocellulosic carbohydrates) is more preferable due to the atom economy and biomass valorization [16,17]. Our recent work showed that molecule oxygen promotes the synthesis of methyl levulinate (ML) from 5-hydroxymethylfurfural, along with the generation of a small amount of DMS during the reaction [18]. In addition, various furanic derivatives, such as furfural, furoic acid, furan, as well as ML and LA could also lead to the co-generation of DMS. These promising results suggested the possibility
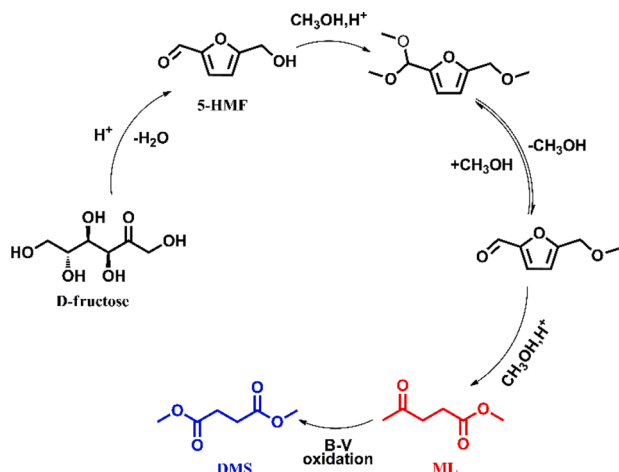
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Scheme 1. Plausible mechanism for the conversion of D-fructose to DMS.
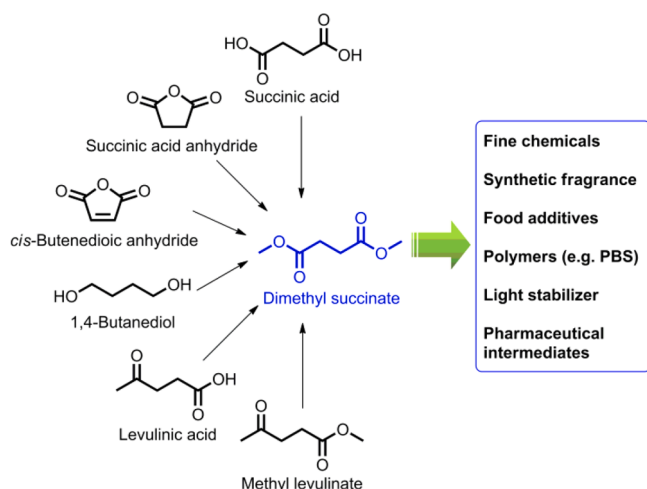

Fig. 1. The synthetic routes of dimethyl succinate and its applications.

of the one-pot synthesis of DMS from furanic compounds, even more appealing from carbohydrates.

In the present study, a metal-free catalytic process for the one-pot synthesis of DMS directly from D-fructose was reported. The catalytic performance of different catalysts and the influence of other experimental parameters, such as temperature, time, oxygen pressure, as well as feedstock variation on the reaction, were investigated in detail. Additionally, the catalyst stability and reaction pathway were also carefully discussed.

1.1. Materials

D-fructose (99% purity), 5-hydroxymethylfurfural (HMF, 98% purity), methyl levulinate (ML, 99% purity), levulinic acid (LA, 99% purity), furfural (99% purity), furfuryl alcohol (FA, 98% purity), furoic acid (99% purity), dimethyl succinate (DMS, 99% purity) and dimethyl fumarate (DMF, 97% purity) were used as purchased from Sigma-Aldrich and without further purification. Sucrose (99% purity), D-xylose (99% purity), dimethyl maleate (DMM, 97% purity), methyl formate (97% purity), dimethoxymethane (98% purity), methanesulfonic acid (99% purity) and p-toluenesulfonic acid monohydrate (98% purity) were purchased from the Innochem Company. The catalysts Amberlyst-15, Amberlyst-36, Amberlyst-70, and Nafion were purchased from the Nanda Synthetic Company.

1.2. Typical experiment

All the experiments were carried out in a 50 mL stainless steel autoclave supplied by the Anhui Kemi Company equipped with a thermocouple and magnetic stirrer (800 rpm). Initially, 75 mg of the catalyst was added to 1.5 mmol of the substrate in 15 mL of methanol. Exactly 10 bar pressure of O_2 was added after purging the reaction thrice. The reaction mixture was stirred and heated to the desired temperature. The target temperature was achieved after 10–15 min at which point the time was set to zero. On completion of the reaction, the stirring was stopped and the autoclave was cooled in the water bath until a temperature below 25°C was attained. The residual pressure was removed before releasing the remaining gas and opening the autoclave lit.

1.3. Product analysis

All the experiments were quantified by gas chromatography (GC). After completion of the reaction, the solution was diluted with methanol to a constant volume of 25 mL, and then 0.5 mL of the solution was taken out and mixed with an equal volume of methanol using 2 mg*mL^{-1} octane as the internal standard. The yield of the target compounds was calculated based on the calibration curves using the following forums:

$$Yield(mol\%) = \frac{Mol_{product}}{Mol_{Initial}} \times 100\%$$

where $Mol_{product}$ is the number of moles of the product formed after the reaction, and $Mol_{Initial}$ represents the number of moles of the starting materials loaded before the reaction.

The product was qualitatively analyzed using the Thermo Scientific TRACE 1310 GC–MS and nuclear magnetic resonance (NMR) spectroscopy.

1.4. Characterization

In order to understand the reason for catalyst deactivation, different catalysts chosen in the study were characterized by Fourier transform-infrared spectrometry using the Nicolet iS50.

Elemental analysis was performed using an EA-1108 CHNS Elemental Analyzer.

The acidity of the catalysts were titrated following the literature [28]. In detail, 0.25 g of the catalyst was placed in 25 mL of 0.1 M solutions of sodium hydroxide. The vial was sealed and shaken for 24 h and then 10 mL of the filtrate was pipetted and the excess of the base was titrated with 0.1 M HCl. The mole acidity of the catalyst was calculated under the assumption that NaOH neutralizes all acidic groups.

2. Results and discussion

2.1. Screening the activity of the catalysts

Our previous study suggested that molecular oxygen could accelerate the conversion of HMF to ML and efficiently remove the *in-situ* formed of humins [18]. Interestingly, 6% of DMS was yielded from HMF on performing the reaction in the presence of O_2 and zeolite H-beta25, which was believed to be formed by the B-V oxidation of ML. Many zeolitic materials are inferior to Amberlyst-15 in B-V oxidation due to the higher acidity of Amberlyst-15 leading to the formation of more peroxidic materials [19]. Moreover, Amberlyst-15 has been successfully used for the B-V oxidation of furfural and levulinate derivatives [6,20, 21]. Consequently, various heterogeneous sulfonic Amberlyst-based catalysts and homogenous sulfonic acid based catalysts were tested for the one-pot conversion of D-fructose into DMS (Fig. 2). In the absence of catalyst, no products were detected by GC and GC–MS spectra. Methane sulfonic acid and Nafion (perfluorosulfonic acid resin) offered a 70% yield of ML with trace amount of DMS. On the other hand, homogeneous
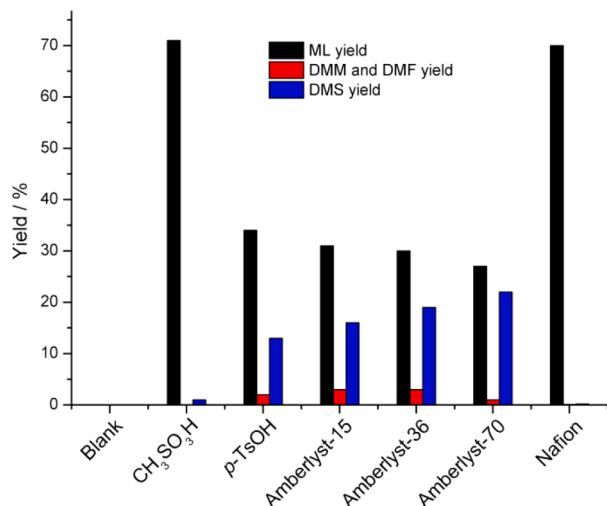
Fig. 2. The effect of catalysts on the conversion of D-fructose.

p-toluenesulfonic acid and all the tested heterogeneous Amberlyst catalysts showed better catalytic performance in terms of DMS production. More than 20% yield of DMS along with 27% of ML was selectively produced using the Amberlyst-70 catalyst at 150°C for 2 h. As a result, a considerable yield of DMS (22%) was obtained from D-fructose *via* one-pot synthesis using the Amberlyst-70 catalyst for the first time, which encouraged further optimization of the process.

Reaction conditions: 1.5 mmol D-fructose, 75 mg catalyst, 15 mL methanol, 10 bar O$_2$, 150°C, 2 h.

2.2. Effect of reaction temperature

The influence of reaction temperature on the conversion of D-fructose was initially studied in the range from 120 to 160°C using Amberlyst-70. As shown in Fig. 3, the yield of DMS exhibited a volcanic trend with the reaction temperature and give a yield of 27% at 130 and 140°C. This suggested that a particular reaction temperature is favorable for the formation of DMS. It was suggested that a lower reaction temperature was not efficient enough for the conversion of ML formed into DMS *via* B-V oxidation. As a result, a high yield of ML (64%) was obtained along with the formation of only 5% of DMS at 120°C. On the other hand, a higher reaction temperature was supposed to lead to poor molecular oxygen solubility in methanol, thus, impeding to the B-V oxidation of ML to DMS. Therefore, 130°C was chosen as the optimized reaction
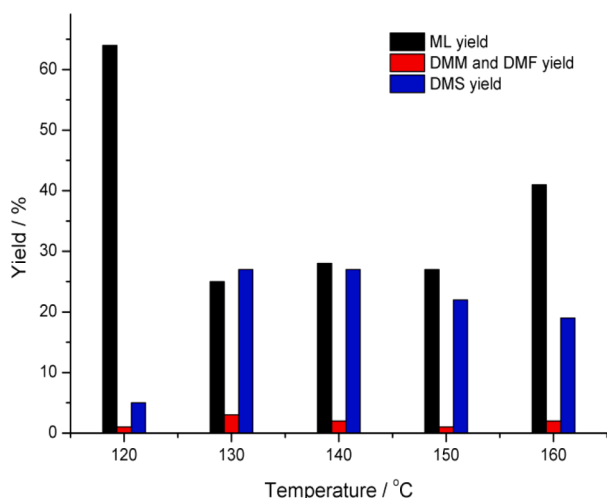
temperature in order to gain a better yield of DMS in the following study.

Reaction conditions: 1.5 mmol D-fructose, 75 mg Amberlyst-70, 15 mL methanol, 10 bar O$_2$, 120–160°C, 2 h.

2.3. Effect of oxygen pressure

Since the formation of DMS is an oxidation process, the effect of oxygen pressure on the conversion of D-fructose was assessed in this study. Fig. 4 shows that an increase in the oxygen pressure initially promoted the yield of ML with no formation of DMS under the oxygen pressure lower than 5 bar at 130°C for 2 h. As the oxygen pressure increased to 10 bar, 27% of DMS was formed, and ML yield was severely decreased from 62% at 5 bar to 26% at 10 bar, indicating the high dependency of oxidation of ML on the oxygen pressure. On further increasing the oxygen pressure, the yields of DMS, DMM and DMF were slightly increased. Considering the yield of DMS and reaction mass balance, 10 bar of oxygen was chosen as the optimal reaction pressure.

Reaction conditions: 1.5 mmol D-fructose, 75 mg Amberlyst-70, 15 mL methanol, 0–30 bar O$_2$, 130°C, 2 h.

2.4. Effect of catalyst loading and reaction time

The influence of catalyst dosage on the conversion of D-fructose with dependence on reaction time was investigated (Table 1). On loading 25 mg of Amberlyst-70, the yields of all the products were limited to a certain extent from 1 to 4 h (Table 1, entries 1–3), suggesting the insufficient amount of catalyst loading for the efficacious conversion of D-fructose. The increase in catalyst loading from 25 to 75 mg significantly improved the yield of ML in a short reaction time (Table 1, entries 4 and 8). Meanwhile, the yield of DMS gradually increased and reached 31% in 4 h under 75 mg of Amberlyst-70 (Table 1, entry 11). On the other hand, the experiments were also carried out under N$_2$ atmosphere (Fig. S1). As expected, the highest yield of ML with a value of 80% was achieved in 2 h with no formation of DMS. Meanwhile, 70% of ML was obtained in 30 min under O$_2$ atmosphere (10 bar), which was higher than that obtained under N$_2$ atmosphere (56%), indicating the increase in the conversion of D-fructose to ML in the presence of molecular oxygen. Moreover, molecular oxygen further participated in the conversion of ML to DMS. Interestingly, Table 1 also revealed that higher values of catalyst loading were unfavorable for the formation of DMS. This is indicated by the absence of DMS formation upon the addition of 100 mg of Amberlyst-70 in 1 h, in contrast to a 12% yield of DMS on the addition of 75 mg of Amberlyst-70 under identical reaction conditions (Table 1, entry 14 vs entry 9).

To understand this unexpected phenomenon, ML was used as the starting material along with different amounts of catalyst (Table 2
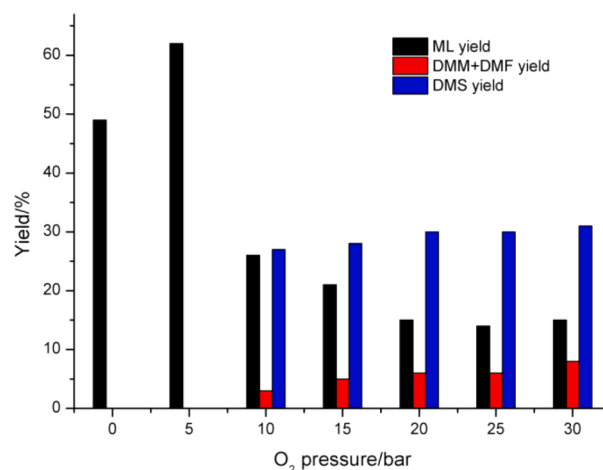

Fig. 3. The effect of reaction temperature on the conversion of D-fructose.

Fig. 4. The effect of oxygen pressure on the conversion of D-fructose.

Table 1

The effect of catalyst loading and reaction time on the conversion of D-fructose.

Entry	Reactant	Amberlyst-70 loading/mg	P_{O_2}/bar	T/°C	t/h	Yield /%		
						ML	DMM+DMF	DMS
1	D-fructose	25	10	130	1	22	8	8
2					2	24	8	11
3					4	23	6	11
4	D-fructose	50	10	130	0.5	50	0	0
5					1	49	5	9
6					2	38	5	12
7					4	16	5	22
8	D-fructose	75	10	130	0.5	70	0	0
9					1	54	2	12
10					2	25	3	27
11					4	23	3	31
12					6	17	2	30
13	D-fructose	100	10	130	0.5	71	0	0
14					1	88	0	0
15					2	33	1	27
16					4	28	1	29
17					6	27	1	28

Reaction conditions: 1.5 mmol D-fructose, 25–100 mg Amberlyst-70, 15 mL methanol, 10 bar O_2, 130°C, 0.5–6 h.

Table 2

The effect of catalyst loading and reaction time on the conversion of ML.

Entry	Reactant	Amberlyst-70 loading/mg	P_{O_2}/bar	T/°C	t/h	ML conv./%	Yield/%	
							DMM+DMF	DMS
1	ML	25	10	130	1	22	0	15
2		50				51	0	39
3		75				59	0	44
4		100				47	0	38
5		75			0.5	8	0	5
6					2	67	0	50
7					4	70	0	50

Reaction conditions: 1.5 mmol ML, 25–100 mg catalyst, 15 mL methanol, 10 bar O_2, 130°C, 0.5–4 h.

entries 1–4). The results were *in line* with that obtained with D-fructose, where a poor DMS yield and ML conversion were observed on adding 100 mg of Amberlyst-70 compared to those achieved using 75 mg of Amberlyst-70 (Table 2, entry 3 *vs* entry 4). The GC–MS analysis of the reactions showed that a new compound, namely methyl 4,4-dimethoxy-pentanoate, was formed on the addition of 100 mg Amberlyst-70 (Fig. S2). Therefore, a higher catalyst loading is speculated to favor the acetalization of ML with methanol, which protects the carbonyl group, and hinders the following B-V oxidation process required to form DMS. The effect of reaction time on the conversion of ML (Table 2 entries 3, 5–7) also revealed that the best yield of 50% was obtained during the synthetic reaction of DMS performed for 2 h with a selectivity of 75%, which was better than that reported with H_2O_2 [6]. Noting that there was no formation of DMM and DMF from ML (Fig. S2), indicating they were probably generated from HMF derivatives as proposed in our previous study [17].

2.5. Screening of the substrate

In order to expand the application scope of this system, different bio-based substrates were investigated for the one-pot and metal-free synthesis of DMS (Table 3). A comparable yield of DMS was obtained from HMF (30%, Table 3, entry 1), and furfuryl alcohol resulted in a better DMS yield of 46%, probably attributed to its easier alcoholysis to form ML (Table 3, entry 2). Interestingly, only the acetalization of furfural was observed under optimal conditions (75 mg Amberlyst-70, 10 bar O_2, 130°C, 2 h). Thus, harsher reaction conditions (150°C, 20 bar O_2) were needed for the production of DMS (42%) from furfural (Table 3, entry 4), which also goes through B-V oxidation similar to that with H_2O_2 [20, 22]. Furoic acid only provided a 22% yield of DMS even at 150°C (Table 3, entry 5). Sucrose and D-xylose were also studied but yielded much lower values (Table 3, entries 6–7). Since the one-pot synthesis of DMS from furfural and furfuryl alcohol has been rarely reported, the considerable obtained yield of DMS is very promising.

Reaction conditions: 1.5 mmol substrates, 75 mg Amberlyst-70, 15 mL methanol, 10 or 20 bar O_2, 130 or 150°C, 2 h. a: the only product is 2-(dimethoxymethyl)furan; b: the major product is methyl furoate.

Table 3

One-pot conversion of different substrates into DMS.

Entry	Reactant	Amberlyst-70 loading/mg	P_{O_2}/bar	T/°C	t/h	Yield/%		
						ML	DMM+DMF	DMS
1	HMF	75	10	130	2	41	4	30
2	Furfuryl alcohol		10	130		27	0	46
3	Furfural[a]		10	130		0	0	0
4	Furfural		20	150		2	0	42
5	Furoic acid[b]		20	150		0	0	22
6	Sucrose		20	150		13	2	13
7	D-xylose		20	150		0	0	6

2.6. Catalyst stability

Catalyst stability is an important factor to assess its catalytic performance in a reaction. However, in this study, all of these heterogeneous catalysts were unexpectedly dissolved in the solution after the reaction, although some of them exhibited a higher operating temperature (Amberlyst-70, max. 190°C, Nafion, max. 350°C). The change in the catalytic activity of the catalyst was studied by repeating the experiments of Amberlyst-70. After each reaction, the solution was collected and evaporated in a vacuum. The residue was washed thrice with ethyl acetate to remove the products with high boiling points, which was then dried at 60°C for the next catalytic reaction. Fig. 5 shows that the catalyst has a poor reusability, as the yields of both ML and DMS decreased from 26% to 17% and 27% to 6% after three cycles respectively. Later, the control experiments were performed to probe the possible reason for the decreased in reactivity.

Reaction conditions of run 1–3: 1.5 mmol D-fructose, 75 mg Amberlyst-70 or recovered catalyst, 15 mL methanol, 10 bar O_2, 130°C, 2 h. Control test 1: 75 mg Amberlyst-70 was pretreated at 130°C for 2 h in presence of 15 mL methanol and 10 bar O_2, followed by adding 1.5 mmol D-fructose and recharging 10 bar O_2. Then the autoclave was reheated to 130°C for 2 h; Control test 2: 75 mg Amberlyst-70 was pretreated at 130°C for 2 h in presence of 15 mL methanol and 10 bar O_2, followed by removing all solvents by vacuum drying. Then 1.5 mmol D-fructose with 15 mL fresh methanol and 10 bar O_2 was recharged, and the autoclave was reheated to 130°C for 2 h.

In control test 1, Amberlyst-70 was pretreated under optimal conditions, and 1.5 mmol D-fructose and 10 bar O_2 were loaded to restart a new reaction. As a result, only low amounts of the desired products were detected. Upon pretreatment of Amberlyst-70, two major side products, namely methyl formate and dimethoxymethane, formed from methanol were successfully identified by GC–MS and NMR. Later, these two commercial side-products were separately added for the conversion of D-fructose under standard reaction conditions (Table S1). The experimental results indicated that the side products exhibited negative effects on the conversion of D-fructose into DMS, especially dimethoxymethane. Also, a better reaction result was obtained by removing methyl formate and dimethoxymethane formed during the pretreatment of Amberlyst-70 and addition of fresh methanol (control test 2). Moreover, the catalytic differences within Run 1 and control test 2 also revealed that the pretreatment of Amberlyst-70 resulted in its catalytic activity loss. Thus, it is easy to conclude that the limited yield of DMS (~30% from D-fructose) is possibly due to the negative effects of the side products and deactivation of the catalyst.

The ATR-IR analysis of the fresh and spent Amberlyst-70 was also performed (Fig. 6). The characteristic vibration bands of the catalysts at 1161 and 1033 cm^{-1} are assigned to the O=S=O stretching vibration
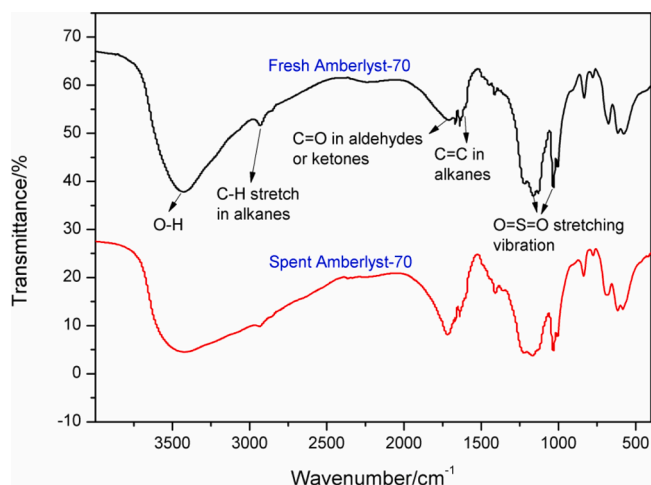

Fig. 6. FT-IR spectra of fresh and spent Amberlyst-70.

group [23]. The apparent vibration changes in the O—H and C=O bands of the spent catalyst demonstrated a partially disintegrated catalyst structure after the reaction. Additionally, the broadened O—H band and strengthened C=O bands implied that the hydrogen-bonding and polarity of the spent catalyst were stronger than those of the fresh one, which might attribute to its dissolution in methanol.

To better understand the changes in Amberlyst-70, p-toluenesulfonic acid, with a comparable catalytic activity with Amberlyst-70 (Fig. 2), was chosen as the model catalyst. After the pretreatment of the p-Toluenesulfonic acid in methanol at 130°C for 2 h under 10 bar O_2, the solvent was evaporated, and the liquid residue was analyzed by recording the ^{1}H NMR spectrum (Fig. S3). Obviously, there are new peaks between 7.5–8.0 ppm ascribed to the hydrogen shift of the benzene ring. The sulfonic acid group might be partially esterified, which is clear evident from the detection of methyl p-toluenesulfonate by GC–MS. In addition, a trace amount of toluene was also detected in the pretreated p-toluenesulfonic acid by GC–MS, suggesting the unstable nature of the sulfonic acid group under the present reaction conditions. The changes in the sulfonic acid group were stipulated to be partially responsible for the catalyst dissolution and deactivation. Furthermore, elemental analysis and acidity measurement of fresh and spent catalysts were performed (Table S2). Unfortunately, only negligible changes in the C, H and S contents within the fresh and spent Amberlyst-70 were observed, suggesting the leaching effect is inconspicuous. The acidity measurement showed unexpectedly a slight increase in the acidity of the spent catalyst, probably attributing to the differences in solubility (acidic groups can be easily and sufficiently titrated or not) [23].

2.7. Plausible reaction mechanism

According to the literature, the one-pot production of ML from D-fructose could be easily realized using an acidic catalyst [24–27]. In this study, ML was indeed observed as the major product under N_2 and O_2 atmospheres (Table 1 entries 4, 8 and 13 at 0.5 h), however, ML was gradually consumed under O_2 atmosphere with time, and DMS was simultaneously formed. This indicated that ML acted as an intermediate for the formation of DMS, which was consistent with the results of the studies [3,6,16]. Additionally, the HMF derivatives were observed at the beginning (Fig. S4). Therefore, a plausible mechanism was proposed based on the results and observations as follows (Scheme 1): Initially, D-fructose was firstly dehydrated into HMF, which was made to undergo etherification and/or acetalization of HMF followed by its alcoholysis to form ML. Finally, the B-V oxidation of ML into DMS was performed in the presence of molecular oxygen and Amberlyst-70. The side products, namely DMM and DMF, were possibly produced *via* the B-V oxidation of HMF, as discussed previously [18].
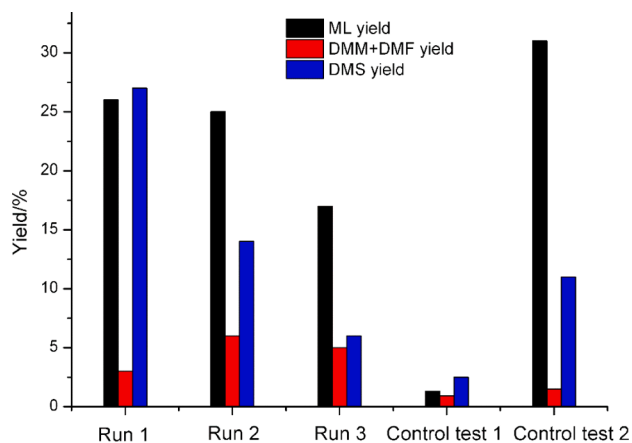

Fig. 5. Reusability of the catalyst on the conversion of D-fructose.

3. Conclusion

In this paper, the one-pot and metal-free synthesis of DMS using Amberlyst-70 catalyst from ᴅ-fructose was reported for the first time. The reaction parameters, such as temperature, time, catalyst dosage, and oxygen pressure, play crucial roles in determining the yield of DMS. Unfortunately, it was found that the catalyst was dissolved in the solution due to its partially disintegrated structure, and deactivation probably by the esterification/leaching of the sulfonic acid groups. It was also demonstrated that the side products, namely dimethoxymethane, generated from the solvent deactivated the catalyst. Further investigation on a more stable and efficient catalyst is being undertaken by our group.

Appendix A. Supplementary data

The Supporting Information is available:

Credit author statement

Liangtu He: Methodology, Investigation, Formal analysis; Lei Liu: Investigation; Yuzhang Huang:Investigation; Xinge Miao: Investigation; Christophe Len: Writing- Reviewing and Editing; Yantao Wang: Conceptualization, Methodology, Writing-original draft; Weiran Yang: Supervision, Writing- Reviewing and Editing, Funding acquisition.

Declaration of Competing Interest

All authors declare that no conflict of interest exists.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mcat.2021.111584.

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