

Hydrolysis of Fruit Waste to Reduce Sugars Using Sulphonated Magnetic Carbonaceous Catalyst

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Abstract: The sulphonated magnetic carbonaceous catalyst ($\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$) was effectively synthesised by functionalising the sulphonic acid group ($-\text{SO}_3\text{H}$) on the surface of the core-shell structure of magnetic nanoparticle grafted activated carbon and analysed by SEM, XRD, and FT-IR. The As-prepared catalyst may aggressively hydrolyse biomass to sugar in a shorter reaction time. Hydrolysis factors such as catalyst dose, reaction temperature, and duration all had a significant impact on the hydrolysis of pomegranate peel waste. The catalyst could liberate 46% of TRS in 2 hours at 140°C from acid-pretreated pomegranate peel waste. This catalyst can be easily regenerated using a magnet and reused for up to three cycles with improved stability.

Key words: Pretreatment, hydrolysis, fruit waste, total reducing sugars, sulphonated magnetic carbonaceous catalyst.

Introduction

The rapid growth of petroleum industrialisation causes the emission of greenhouse gases which increases the demand for clean energy worldwide (Zhang et al., 2017). Such demand kindled the global community to develop environmentally sustainable processes in a greenway. In these contexts, exploring bioethanol from lignocellulosic biomass has been considered a promising alternative to petroleum-based resources. Moreover, it reduces the dependency on fossil resources and fulfills energy scarcity by replacing fossil fuels with renewable resources (Lai et al., 2011; Zhao et al., 2011). Since it possesses beneficial advantages such as being clean and renewable, the utilisation of bioethanol lessens

the CO_2 emissions concerning global warming (Tian et al., 2010).

Lignocellulosic biomass is regarded as carbon-neutral, economical, and possesses potential for biofuel production (Ansanay et al., 2017). It is comprised of cellulose, hemicellulose, and lignin. Among that, cellulose is considered as most readily available, abundant, and sustainable material on earth (Khan et al., 2018). The transformation of cellulose into polysaccharides and monosaccharides seems to be challenging since it is a densely packed structure and insoluble in water (Ren et al., 2013). In these regards, the pretreatment process disrupts the rigid complex structure of biomass to liberate the carbohydrates into cellulose and hemicelluloses. Hydrolysis is a

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simple process that converts carbohydrates into sugar monomers either by homogeneous catalyst (Shadbahr et al., 2017).

Generally, acidic hydrolysis (Tizazu and Moholkar, 2018) and enzyme-driven hydrolysis (Talekar et al., 2018) are the most used techniques to produce reducing sugar. Even though it is highly efficient, these techniques have brought significant drawbacks, especially in terms of difficulty in catalyst recovery, high cost of the enzyme, wastewater generation, and reactor corrosion (Fang et al., 2011; Zhang and Fang, 2012). With the focus on eliminating these issues, much effort has been investigated on the development of a new strategy by heterogenizing the homogeneous catalysts. For instance, Intaramas et al. (2018) reported the transformation of cassava mash to sugar monomers using a solid acid catalyst by thermo-hydrolysis at 140°C for 6 hours.

Lately, Deng et al. (2016) investigated the pre-hydrolysis liquor of corncob using biochar catalyst for furfural production. In addition, the $-\text{SO}_3\text{H}$, phenolic $-\text{OH}$, and COOH groups bearing biochar catalyst were prepared by carbonisation of corncob followed by functionalisation with acid sites. However, it is still required to develop a new catalyst with easy separation and catalyst reusability for better carbohydrate conversion.

In recent years, the synthesis of catalyst with magnetic properties has attracted researchers and is widely employed in various applications. Owing to their self-interaction, they can be used as catalyst support as they are readily aggregated. Encapsulating the magnetic nanoparticles with the carbon shell will provide shielding to the core during sulphonation, thereby diminishing the risk of magnetic core corrosion (Yuan et al., 2015). Also, surface functionalisation with sulphonic groups on the carbon shell is a prospective way to construct a bridge between homogeneous and heterogeneous catalysts, which will construct an acid catalyst (Koukabi et al., 2012). Undoubtedly, using a heterogeneous catalyst for biomass conversion is an effective way, and offers easy separation and catalyst recyclability. In the previous attempt, the magnetic carbon acid catalyst hydrolysed the pomegranate peel waste at 140°C for 2 hours, recycled the catalyst up to three times, and then, the catalytic hydrolysate was fermented into bioethanol (Hemalatha and Brinda Lakshmi, 2020).

Pomegranate peel waste (PPW) contains more than 70% cellulosic sugars (Abid et al., 2017), which can be used as an appropriate substrate for catalytic hydrolysis.

After fruit processing, the remaining residues were used as the potential resource to produce biofuel and biochemicals including alkanes, organic acids, lipids, and so forth. Processing this waste and turn it into energy could make the agro-processing sector more competent and support the rural livelihoods (Demiray et al., 2019; Nizami et al., 2017).

In this work, a synergistic sulphonated magnetic carbonaceous catalyst was well-designed with a core-shell structure ($\text{Fe}_3\text{O}_4@\text{AC}-\text{SO}_3\text{H}$), as an environmentally benign and efficient catalyst, which efficiently hydrolysed the biomass into reducing sugars in aqueous medium. Furthermore, the sulphonated magnetic carbonaceous catalyst was easily recovered through external magnetic separation.

Experimental Section

Feedstock and Pretreatment

Pomegranate peel waste (PPW) was collected locally in Chennai, Tamil Nadu, India. PPW was chopped into small pieces with an even size, washed with de-ionised water and oven-dried. Dried PPW was grinded, and then sieved through 80 mesh. Pretreatment was conducted using dilute acid (0.25% v/v H_2SO_4 at 120°C for 1 h) with solid loading of 10% (w/v). After pretreatment, the biomass slurry was washed for few times with distilled water until it became neutral. The residue was then oven-dried at 70°C and stored in a container at room temperature. The composition analysis was carried out and was reported in the previous literature (Hemalatha and Brinda Lakshmi, 2020).

Catalyst Preparation

The sulfonated magnetic carbonaceous catalyst was synthesised as follows. For a typical procedure, magnetic nanoparticles (300 mg) and activated carbon (2 g) were mixed in 50 ml of distilled water and sonicated for 30 minutes. Then, the reaction mixture was heated at 100°C under vigorous stirring in an oil bath to attain complete vapourisation and, the solid residue was then heated to 200°C for 1 h. The resulting solid $\text{AC}/\text{Fe}_3\text{O}_4$ was then sulfonated using H_2SO_4 with a 1:10 ratio in an oil bath and heated to 150 °C for 3 h to introduce $-\text{SO}_3\text{H}$ groups on the surface catalyst. Afterward, the suspension was allowed to cool down to room temperature and washed with hot water three times until no sulphate ions were detected in the aqueous wash. Finally, the resulting product was dried in a hot air oven at 60°C for 12 h and it was denoted as $\text{Fe}_3\text{O}_4@\text{AC}-\text{SO}_3\text{H}$.

Catalyst Characterisation

Scanning electron microscopy (SEM) images were recorded with VEGA3, TESCAN model microscope at 30 kV accelerated voltage. The X-ray diffraction (XRD) pattern of the sample was determined using Bruker (D8 Advance), equipped with a Cu K α radiation source ($K=1.54\text{\AA}$). FT-IR measurement was recorded using a JASCO model instrument in the wave number range of 500 to 4000 cm^{-1} .

Biomass Hydrolysis

Catalytic hydrolysis of pretreated PPW was conducted in a Teflon-lined stainless-steel autoclave (50 ml) under various conditions. Before hydrolysis, the feedstock was dispersed in deionised water and ultrasonicated for 15 minutes at 38°C with a frequency of 50 Hz, and the catalyst was added to the mixture. The reaction mixture was subjected to heating to the desired temperature for a certain period. After completion of the reaction, the spent catalyst was recovered with the aid of an external magnet, washed with distilled water, and dried at 70°C. The supernatant was collected and centrifuged to 3000 RPM. The hydrolysate was analysed for the existence of total reducing sugars (TRS) by the DNS (3,5-dinitrosalicylic acid) method. The total reducing sugar (TRS) in hydrolysate was measured by the DNS method (Hu et al., 2016).

Regeneration and Re-use of Catalyst

Finally, the spent catalyst was regenerated with the assistance of an external magnet. Then, it was washed with deionised water and oven-dried overnight at 60°C. The spent catalyst was reused for the next consecutive runs under the optimised conditions to determine the stability of the catalyst. The TRS yield was screened for each consecutive run.

Results and Discussion

Catalyst Characterisation

SEM Analysis

The catalysts obtained were analysed using the scanning electron microscopic images obtained for the catalysts before and after the sulphonation process. The images indicate the particle morphology of the catalysts. Also, from the images (Figure 1a) the aggregated morphology in the activated carbon-coated Fe_3O_4 particles using the hydrothermal route could be observed. Meanwhile, the sulphonated catalyst (Figure 1b) indicates the collapsed porosity, which shows the significant physical changes

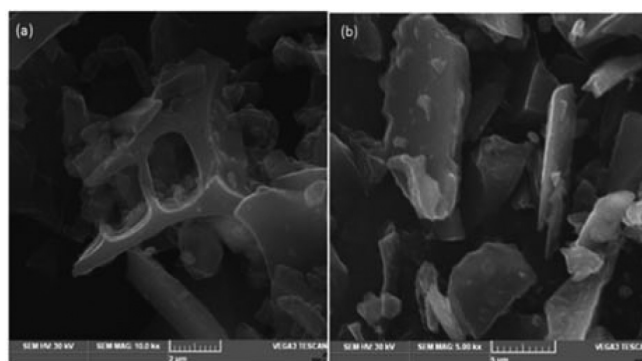


Figure 1: SEM for sulphonated magnetic carbonaceous catalyst (a) before sulphonation $\text{AC/Fe}_3\text{O}_4$ and (b) after sulphonation ($\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$).

on the carbon surface. It is concluded that $\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$ could have potential advantages in biomass hydrolysis.

XRD Analysis

The X-ray diffraction patterns of $\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$ were recorded to study the structural properties (Figure 2). The obtained diffraction pattern proved the structural information of the magnetite nanoparticles remains after sulphonation confirmed by the standard JCPDS card number 19-629. The diffraction peak of magnetic nanoparticles at $2\theta = 37, 53,$ and 63° correspond to (311), (422), and (511) indicating the Bragg reflection of face-centered cubic lattice, respectively (Yuan et al., 2015). Also, the prominent lattice plane (002) confirms that the presence of carbon was in the amorphous region which lies in between the 2θ range of 10° - 30° and agglomerated with the magnetite nanoparticles (Qiu et al., 2018; Zhang et al., 2017). The presence of (001) peak at 45° indicated the formation of aromatic carbon during sulphonation (Shen et al., 2013).

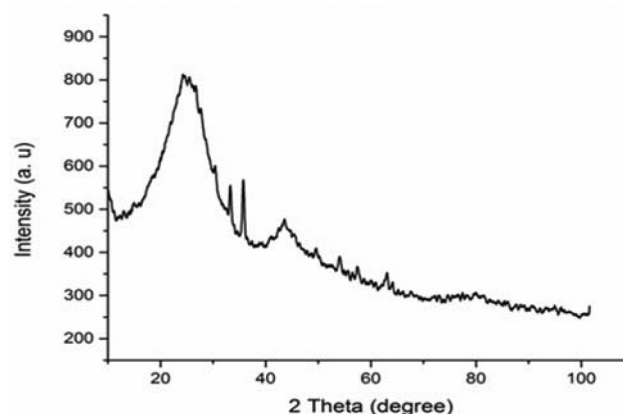


Figure 2: XRD pattern of sulphonated magnetic carbonaceous catalyst ($\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$).

FT-IR Analysis

The surface chemistry of the prepared catalyst was further investigated with FT-IR spectroscopy as shown in Figure 3. The absorption band at around 1154.55 cm^{-1} (S=O symmetric stretching vibration) and 662.52 cm^{-1} (C-S stretching vibrations) indicates that the catalyst was well-sulfonated at 150°C to introduce sulfonic acid groups on the catalyst surface (Peng et al., 2017; Qiu et al., 2018). Moreover, the appearance of the vibration peak at around 3321.67 cm^{-1} belongs to the O-H stretching which indicates the existence of the phenolic OH group. The peak at 547.50 cm^{-1} belongs to the stretching vibration of Fe-O (Su et al., 2015). As a result, these experimental results confirmed that acid sites were grafted successfully onto the surface of carbon channels over the magnetic core.

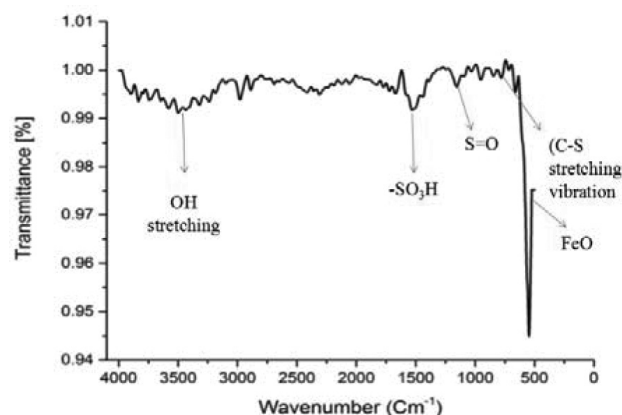


Figure 3: FTIR spectra of sulphonated magnetic carbonaceous catalyst ($\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$).

Biomass Hydrolysis

Effect of Catalyst Dosage

The effect of catalyst dosage was studied on pretreated PPW to attain the maximum reduction of sugar. Figure 4 illustrates the effect of catalytic activity on pretreated biomass. It is noticed that the increase in catalyst loading increases the TRS yield. The maximum TRS yield of 27% was observed at the 1 h of reaction time using 100 mg of catalyst. The increase in TRS yield could be attributed to the increase of active sites on the catalyst surface (Xiong et al., 2014; Yin et al., 2015), which facilitates hydrolysis more effectively. Then, it is noted that the TRS yield was found to decrease due to the excessive catalytically active sites, which leads to glucose degradation. As discussed above, the excessive catalyst dosage leads to the formation of toxic byproducts including 5-HMF, levulinic acid, and humins, which probably accelerated the main reaction

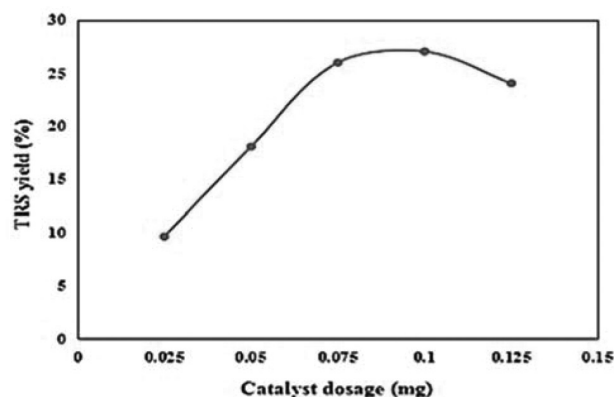


Figure 4: Effect of catalyst dosage on total reducing sugar (TRS) yield (Reaction Conditions: 50 mg of pretreated PPW, 1 h, 130°C).

into unexpected byproducts (Yang et al., 2015). Hence, 100 mg of catalyst dosage was set to be optimal.

Effect of Temperature

Figure 5 shows the effect of temperature on PPW biomass hydrolysis with a sulphonated magnetic carbonaceous catalyst. The yield of TRS was found to be significantly higher with the increase of temperature from 120 to 140°C , reaching 35%. In accordance with these results, it is noted that the increase in TRS yield is attributed to the molecules overcoming the physical barrier of activation energy to move freely at a higher temperature, which could accelerate the fracture of glycosidic bonds of cellulose effectively while interacting with the active sites on the catalyst (Hu et al., 2016). However, the TRS yield seemed not to be influenced by the further elevation of reaction temperature, and it was reduced at 150°C and above. At higher temperatures, this might result in the fast

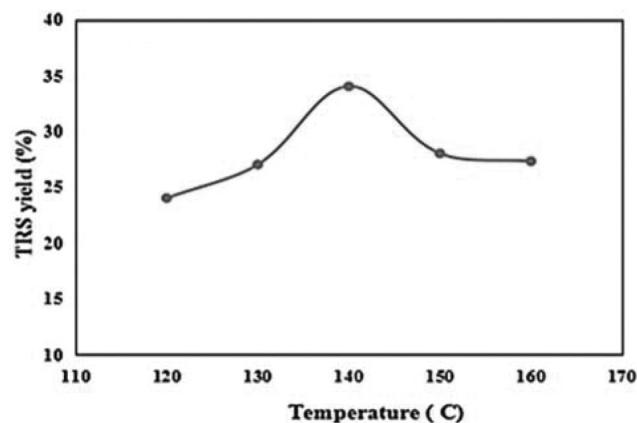


Figure 5: Effect of temperature on total reducing sugar (TRS) yield. (Reaction conditions: 50 mg of pretreated PPW, 1 h, 100 mg of catalyst).

breakdown of produced reducing sugar into various by-products (Su et al., 2020; Wang et al., 2014). Hence, the optimised reaction temperature is found to be 140°C.

Effect of Reaction Time

The influence of reaction time on biomass hydrolysis was investigated as shown in Figure 6. In the case of using a sulphonated magnetic carbonaceous catalyst for biomass hydrolysis, the TRS yield starts to increase linearly with an increase in reaction time and reaches the maximum yield of 46% at 120 min. After 120 min, the TRS yield dropped to 40%. The increase in reaction time was greatly inducing the rate of hydrolysis to the higher (Tong et al., 2013). The reduction in TRS yield is due to the sugar decomposition. This phenomenon should be attributed to the fact that the higher temperature will degrade the sugar through the intermolecular condensation reaction leading to other by-products formation such as furan (Guo et al., 2013; Nata et al., 2015). Hence, the optimal reaction time is 120 min and was chosen as the appropriate value and it was compared with other reported works of literature in Table 1.

Stability of Catalyst

In order to assess the stability and reusability of the catalyst, the performance of the catalyst was studied and illustrated in Figure 5. It could be seen from

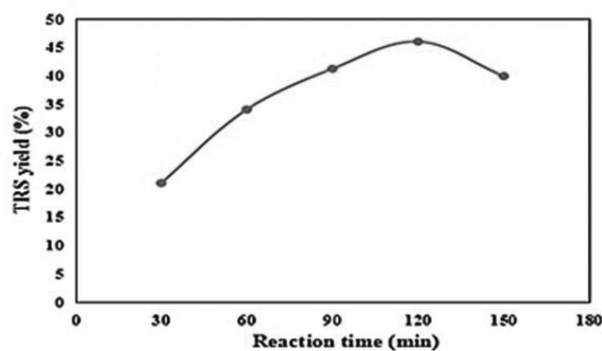


Figure 6: Effect of reaction time on total reducing sugar (TRS) yield. (Reaction conditions: 50 mg of pretreated PPW, 140°C, 100 mg of catalyst).

the figure that the catalyst can be recycled from the reaction system and reused up to three runs. In the first experiment, 46% of the TRS yield was achieved and the catalyst was separated with the aid of an external magnetic field. The results showed the prominent stability of the spent catalyst in the second run with 41% retained, while in the third run, it was reduced to 32%. The activity of the recycled catalyst was declined, probably due to some loss of catalyst (Guo et al., 2012). After hydrolysis, the heterogeneous catalyst was recovered magnetically and can be reused for the next

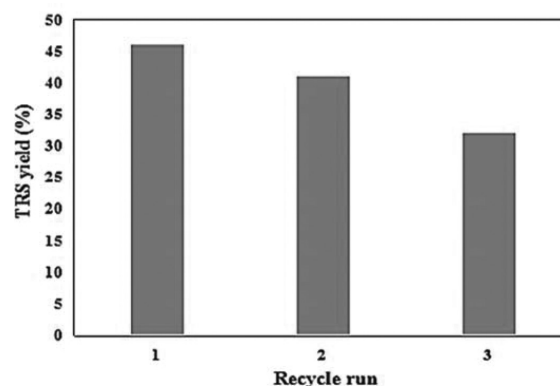


Figure 7: Recyclability of magnetic carbon acid catalyst ($\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$). Reaction conditions: 50 mg of biomass, 140°C, 2 h, 150 mg of catalyst).

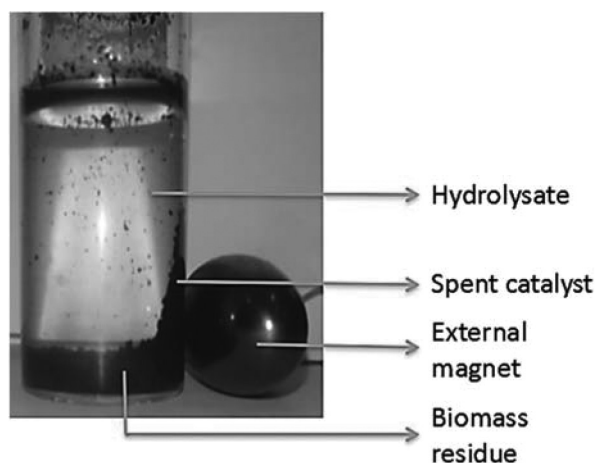


Figure 8: Magnetic property of $\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$.

Table 1: Comparison of hydrolysis of PPW using a prepared catalyst with other heterogeneous catalyst

S.No	Solvent	Biomass	Catalyst	Conditions	TRS Yield (%)
1	[BMIM]Cl	Cellulose	MLC-SO ₃ H	140 °C, 150 min	69.3
2	[BMIM]Cl	Cellulose	Fe ₃ O ₄ @C-SO ₃ H	110 °C, 3 h	72.1
3	Water	Cellulose	H ₃ PW ₁₂ O	180 °C, 2 h	54
4	[BMIM]Cl	Cellulose	Fe ₃ O ₄ @SiO ₂ -SO ₃ H	130 °C, 8 h	73.2
5	Water	Pomegranate peel waste	Fe ₃ O ₄ @AC-SO ₃ H	140 °C, 2 h	46 (This work)

run. As a result, the magnetic material grafted catalyst overruled the hydrolysis reaction and showed their advantages could demand to take a role in biorefineries (Figures 7 and 8).

Conclusion

As shown here, catalytic conversion of cellulosic biomass for sugar leaching over catalyst species grafted magnetic nanoparticles becomes trending in the context of developing an alternative way for energy demand. The acid pretreatment was found to be an effective approach to break down the complex structure of PPW. The magnetic carbon acid catalyst was employed for the one-step conversion of biomass, and the 46% high yield of TRS was achieved with 100 mg of catalyst dosage at a relatively moderate temperature of 140°C for two hours. Moreover, $\text{Fe}_3\text{O}_4@\text{AC-SO}_3\text{H}$ could be collected magnetically from hydrolysate and reused for up to three runs. It is concluded that the prepared catalyst could be an interesting option for biomass hydrolysis and also offers the advantages of lower operational cost, easy separation and pollution-free.

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