

Sulfonated magnetic carbon nanoparticles from eucalyptus oil as a green and sustainable catalyst for converting fructose to 5-HMF

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ABSTRACT

Sulfonated magnetic carbon nanoparticles (SMCNs) were synthesized from eucalyptus oil via co-pyrolysis with ferrocene and sulfonation by H₂SO₄. Catalytic performance of SMCNs for conversion of fructose to 5-hydroxymethylfurfural was examined within a designated range of reaction temperature (120–180 °C) and time (30–240 min). 84% conversion of fructose and 51.6% yield of 5-hydroxymethylfurfural could be achieved with catalyst-to-fructose mass ratio of 0.167 at 180 °C and the reaction time of 30 min. The magnetic property of SMCNs also facilitated recovery and recycling of the spent catalyst. Additionally, more than 50% yield of 5-hydroxymethylfurfural could be achieved after three cycles.

1. Introduction

Biomass exhibits potential as a renewable and economical feedstock to substitute fossil fuel [1,2]. Cellulose is one kind of biomass employed for producing biofuel and various platform chemicals including 5-Hydroxymethylfurfural (5-HMF). 5-HMF draws many research interests due to its promising applications as additive in the biofuel production, coating agent and pharmaceutical products [3]. Generally, 5-HMF could be produced from dehydration and hydrolysis of cellulose or sugar. Typical processes for converting biomass into 5-HMF strongly depend on effective catalyst usage. Additionally, conversion of biomass to 5-HMF could be achieved with the acid-catalyzed hydrolysis following by dehydration. Because the chemical structure of ketohexose (i.e. fructose) is more reactive when compared to aldohexose (i.e. glucose), conversion of ketohexose to 5-HMF is recognized as an efficient mean. However, most of catalysts employed for ketohexose conversion were normally homogeneous aqueous catalyst, i.e. sulfuric acid [4]. Main disadvantage of homogeneous catalyst is difficulty in its separation, and corrosion because of its homogeneity. Therefore, many heterogeneous catalysts have become a pertinent research issue for effective production

of 5-HMF from ketohexose.

Among various heterogeneous catalysts, carbon nanoparticles (CNPs) are recognized as a promising candidate for catalytic conversion of biomass owing to effect of high porosity and designated functional groups on their surface. In comparison with other CNPs, magnetic carbon nanoparticles (MCNs) exhibit some superior characteristics, i.e. easy handling, high surface area, and effective functionalization. Accordingly, exploration of functionalization of CNPs has been proposed and investigated by various research teams [4–6]. CNPs have been employed in conversion of biomass into platform chemicals owing to their thermal stability, high surface area and flexible functionalization via numerous methods [7]. Based on its particulate form, such CNPs could be simply recovered after the reaction was terminated.

Conversion of glucose, fructose or cellulose via dehydration and hydrolysis using carbonaceous catalysts has been explored under various conditions [8]. However, clear understanding in effect of operating variables, especially reaction temperature and time has still been an important issue for many researchers [9,10]. In this work, magnetic carbon nanoparticles (MCNs) were synthesized from eucalyptus oil as a bio-renewable feedstock with the presence of ferrocene. Then grafting

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with sulfonic group could provide sulfonated magnetic carbon nanoparticles (SMCNs) as heterogeneous catalyst for converting fructose to 5-HMF. Both resultant MCNs and SMCNs possess magnetic properties due to the presence of Fe within their nanostructure. Para-magnetism remained in SMCNs even after sulfonation, suggesting the potential of their recovery and recycling. Examination on catalytic performance of SMCNs for converting fructose to 5-HMF in a batch autoclave reactor under designated conditions of reaction temperature, reaction time, and SMCN loading was the objective of this work. Reusability of regenerated SMCN catalysts was also examined.

2. Experimental

2.1. Material

Eucalyptus oil was purchased from Peerasuk Chemical and Package (Thailand). Ferrocene (98% Fe) and Fructose (≥ 99 wt%) were obtained from Sigma-Aldrich (Germany). Hydrogen peroxide (30 wt% H_2O_2) and sulfuric acid (95–97 wt%) were obtained from Qrec (France). All chemicals were used without additional treatment or purification.

2.2. Synthesis of sulfonated magnetic carbon nanoparticles

MCNs were prepared via co-pyrolysis of eucalyptus oil and ferrocene in a quartz tube reactor equipped with an electrical furnace as illustrated in Fig. S1. In short, eucalyptus oil and ferrocene would be thermally decomposed, resulting in generation of carbon and iron atomic clusters. Condensation of iron nanoparticles would catalyze the self-assembly of carbon nanostructures covering such iron nanoparticles [11]. Concentrated sulfuric acid was added into the as-synthesized MCNs within a Teflon-sealed autoclave reactor at 150 °C for 600 min. The sulfonated sample was rinsed repeatedly with hot DI water until neutral. Finally, sulfonated magnetic carbon nanoparticles (SMCNs) were dried and stored in a desiccator.

2.3. Characterization of synthesized product

Fourier-transform infrared (FTIR) spectroscopy (FTS 6000 FTIR spectrometer, Bio-rad, USA) was employed for characterizing functional groups existing on the surface of the resultant SMCNs. Scanning electron microscope with elemental analysis (SEM-EDX, Hitachi, S-3500 N, Japan) was used for morphological analyses of the pristine MCNs and SMCNs. Brunauer-Emmett-Teller (BET) specific surface area of all resultant samples was measured using Belsorp-mini II (Bel Japan, Japan) with liquid nitrogen adsorption method.

2.4. Catalytic test

A designated amount of fructose and SMCNs were mixed in 30 mL of DI water and loaded into a Teflon-coated container in a high-pressure hydrothermal autoclave reactor equipped with temperature controller. The autoclave reactor was filled with N_2 until the reactor pressure achieved 15 bar at room temperature. Catalytic performance of SMCNs was investigated with respect to reaction time (30–240 min), reaction temperature (120–180 °C), catalyst-to-fructose ratio (0.083; 0.125; 0.167; 0.208 g.g^{-1}). After each reaction, the mixture was immediately quenched and then collected from the reactor. Spent catalyst was separated from the mixture using an external magnet to get the liquid sample for analysis. Chemical composition of liquid samples was analyzed by a High-Performance Liquid Chromatography (HPX-87H sugar 300 \times 7.8 mm, Bio-Rad, Hercules, US.). Fructose conversion was evaluated based on initial amount (F_i) and remaining amount (F_f) as described in Eq. (1). Meanwhile, yield of 5-HMF together with levulinic acid and furfural was determined by Eqs. (2), (3), and (4) based on molar amount of each substance detected by HPLC analyses.

$$\text{Fructose conversion (mol\%)} = \frac{F_i - F_f}{F_i} \times 100\% \quad (1)$$

$$5\text{-HMF yield (mol\%)} = \frac{\text{mole of produced 5-HMF}}{F_i} \times 100\% \quad (2)$$

$$\text{Levulinic acid yield (mol\%)} = \frac{\text{mole of produced LA}}{F_i} \times 100\% \quad (3)$$

$$\text{Furfural yield (mol\%)} = \frac{\text{mole of produced FA}}{F_i} \times 100\% \quad (4)$$

3. Results and discussion

3.1. Characterizations of MCNs and SMCNs

Neutralized samples of resultant MCNs and SMCNs were subjected to SEM-EDX for morphological and elemental analyses. As shown in Fig. S1 (a) and (b), carbon nanotubes and certain amount of carbon nanostructures were observed in both pristine MCN and SMCN samples. Typical SEM image of the SMCN sample revealed a less amount of agglomerates of carbon nanostructures on the surface of carbon nanotubes when compared to those of the MCN sample. Difference between MCNs and SMCNs would be ascribed that treatment by sulfuric acid could remove some portions of amorphous carbon content. Elemental compositions of pristine MCNs and SMCNs are summarized in Table 1 in comparison to other sulfonated carbon materials. These results would confirm that sulfonic group was attached to the surface of carbon nanotubes and carbon nanostructures. The amount of sulfonic groups grafted onto SMCNs could be gauged due to the sulfur content reacting with substrate [15]. After sulfonation, the sulfur content detected on the surface of the SMCN samples was increased to 1.12%. SO_3 group could anchor onto the surface of carbonaceous nanostructure containing defects due to the sp^2 hybridization [6,16].

Generally, active proton (H^+) existing in the acidic condition would react with unstable amorphous carbon content within the as-synthesized MCNs. The removal of such amorphous carbon would result in formation of 'cavity' on the surface of the acid-treated sample [17]. With concentrated sulfuric acid (97%), reactive H^+ would attack the surface of MCNs, resulting in formation of mesoporous structure. Specific surface area and total pore volume of typical MCN and SMCN samples were determined using BET analyses. As summarized in Table 1, the specific surface area of the SMCN ($68.5 \text{ m}^2/\text{g}$) was significantly higher than that of the pristine MCN ($57.6 \text{ m}^2/\text{g}$). These analytical results indicate the presence of a higher portion of mesopore in the MCNs and SMCNs which was confirmed by the nitrogen (N_2) adsorption-desorption isotherm. The porous structure of both MCNs and SMCNs was a typical IV type with a hysteresis loop of H3 type (Fig. S2). A detectable increase in the specific surface area and mesopore of the SMCNs after sulfonation process was attributed to sulfonation time, temperature and acid concentration [6,18]. Change of pore size distribution and surface defects in the SMCNs could be induced by oxidative destruction of carbon bonds in the graphene layers on the surface of the MCNs under intense acid treatment [16].

Fig. 1 is an evidence of functionalization of MCNs to produce SMCNs by sulfuric acid treatment. FTIR spectra of the pristine MCNs and SMCNs reveal the characteristic peaks of C=O stretching at 1725 cm^{-1} and skeleton vibration of the C=C bond of graphitic domain at 1560 cm^{-1} [19]. Additionally, the existence of sulfonic acid group (SO_3) on the surface of the SMCN could also be confirmed with the appearance of the band at 1040 and 860 cm^{-1} , which was in a good agreement with the abovementioned elemental analyses [19,20].

Table 1
Elemental and BET analyses of various sulfonated carbon materials.

Catalyst	Elemental composition (%)				BET analysis		Reference
	C	O	S	Fe	Surface area (m ² /g)	Total pore volume (cm ³ /g)	
Coconut shell biochar	44.31	–	–	–	–	–	[12]
Sulfonated coconut shell biochar	23.39	67.85	6.78	–	99.97	0.002	
Sawdust biochar	68.5	27.44	–	–	2.35	0.001	[13]
Sulfonated sawdust biochar	28.98	61.47	6.62	–	3.3	0.005	
Food waste biochar	75.42	22	–	–	135.2	–	[14]
Sulfonated food waste biochar	69.58	24.81	3.02	–	6.9	–	
Magnetic carbon nanoparticles	89.5	1.3	–	9.2	57.6	0.18	This study
Sulfonated magnetic carbon nanoparticles	88.4	5.5	1.12	5.0	68.5	0.25	

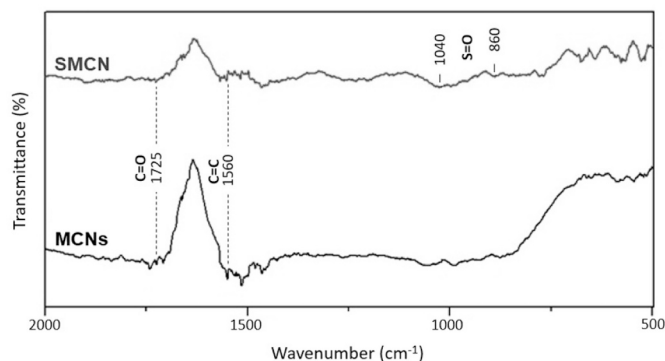


Fig. 1. FTIR spectra of typical samples of MCNs and SMCNs.

3.2. Catalytic conversion of fructose to 5-HMF

3.2.1. Effect of reaction time

In a batch dehydration of fructose, hydroxyl groups would interact with sulfonic groups, resulting in formation of 5-HMF and water molecules [21]. An important issue is that grafting the sulfonic group on the surface of stable SMCNs could provide possibility for keeping the sulfonic group stable until most of fructose could be dehydrated when the reaction time proceeds. The as-prepared SMCNs were used to convert fructose to 5-HMF in aqueous media (de-ionized water) at 180 °C within the reaction time range of 30–240 min. As summarized in Fig. 2, a high

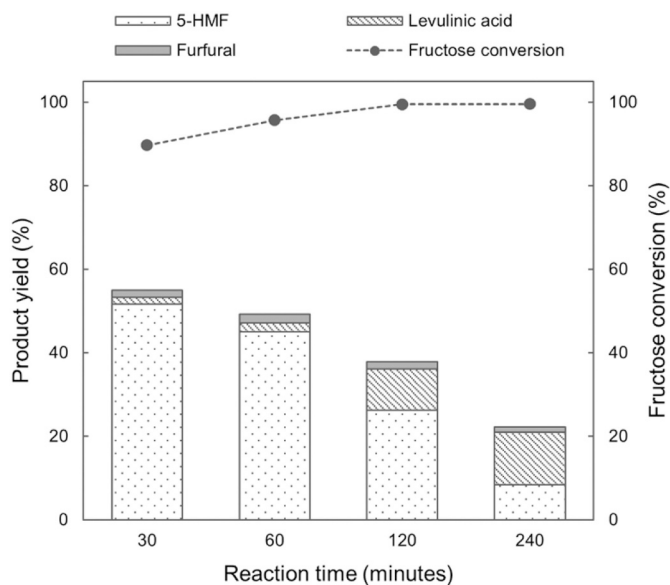


Fig. 2. Effect of reaction time on fructose conversion and product yield (Conditions: 1.2 g fructose, 0.2 g SMCN, 30 mL DI-water, 180 °C).

conversion of fructose of 84% could be achieved within 30 min along with 5-HMF yield of 51.6%. Accordingly, the fructose conversion reached almost 100% within 120 min. Therefore, extension of the reaction time would not further exert effect on the fructose conversion. Despite the higher conversion of fructose, a decrease in 5-HMF yield was oppositely confirmed when the reaction time was extended from 30 to 240 min. The lowest 5-HMF yield of 8.4% was confirmed at the reaction time of 240 min, owing to the 5-HMF rehydration at high temperature [16].

According to our analyses, SMCNs could exert the highest catalytic conversion of fructose into 5-HMF within 30 min, resulting in the highest 5-HMF yield with only low content of levulinic acid (LA) and furfural. An increment in yield of LA from 1.7% to 12.6% was ascribed to rehydration of 5-HMF to LA [10]. Normally, dehydration of fructose to 5-HMF is followed by rehydration of 5-HMF to LA, furfural and possibly formic acid [22]. When water is used instead of other non-polar solvents, interaction between HMF, intermediates and water molecules could favour further hydrolysis of HMF to levulinic acid and formic acid [23]. Without catalyst, formation of undesired by-products would be regulated by polymerization and condensation of 5-HMF at high temperature and extended reaction time [5,6]. Based on HPLC analyses, no detectable amount of such undesired by-products would confirm the promising performance of the SMCN catalysts. The catalytic performance of SMCNs was compared with other solid acid catalysts in term of fructose conversion and product yield (Table 2). Dimethyl sulfoxide (DMSO) and nitromethane could lead to significant conversion of fructose and high yield of HMF at low temperature [26,27]. Meanwhile, the use of water as reaction medium has been explored intensively due to its eco-friendly and low-cost properties. In this work, fructose conversion of 84% and HMF yield of 51.6% (at 180 °C) would be remarked as a significant finding when compared with other previous studies. This consistent result would confirm the potential application of SMCNs as catalyst with the presence of water for biomass conversion.

3.2.2. Effect of reaction temperature

Based on Arrhenius' concept, reaction temperature exerts a significant influence on fructose conversion [5]. Different reaction temperatures were investigated for catalytic conversion of fructose into 5-HMF while reaction time and catalyst loading were fixed at 30 min and C:F ratio of 0.167. A significant increase in both fructose conversion and 5-HMF yield along with the elevation of temperature could be observed in Fig. 3. At 120 °C, only 3.2% of fructose was converted and 0.37% of 5-HMF was obtained. At 150 °C, the catalytic performance was still moderate with 18.6% conversion and 12.7% yield of 5-HMF. A further increment of reaction temperature to 180 °C provided remarkable change in fructose conversion and 5-HMF yield of about 80.3% and 45.4%, respectively. Only a small amount of LA and furfural was obtained at the elevated temperature because they were inevitably formed by the acid-catalyzed rehydration of 5-HMF [13]. Such repeated results indicated the important role of reaction temperature, hence temperature controlling during catalytic conversion of fructose to 5-HMF is crucial. Accordingly, further investigation on the SMCN catalytic performance was conducted at 180 °C.

Table 2
Catalytic dehydration of fructose (F) to HMF using various catalysts (C).

Catalyst	C:F ratio (g.g ⁻¹)	Solvent	Temperature (°C)	Conversion (%)	Yield (%)	Reference
Sulfonated biochar	0.5	Water	180	72	42.3	[24]
Zeolite	0.4	DMSO/Water	160	72.4	49.2	[25]
Sulfonated organosilica	0.1	Water/ Nitromethane	140	93	75	[26]
Carbon-based solid acid	0.8	DMSO	130	99	91.2	[27]
Sulfonated magnetic carbon nanoparticles	0.167	Water	180	84	51.6	This study

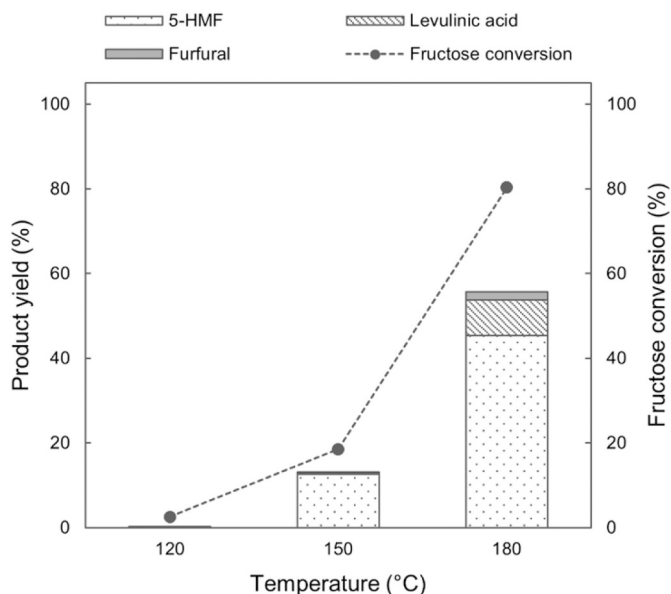


Fig. 3. Effect of reaction temperature on fructose conversion and product yield (Conditions: 1.2 g fructose, 0.2 g SMCN, 30 mL DI-water, 30 min).

3.2.3. Effects of catalyst-to-fructose ratio

Availability of active sites with acidity of heterogeneous catalyst is particularly corresponding with conversion and product yield [28]. Therefore, difference in SMCN loading could certainly affect the fructose conversion [29]. Experimental investigation with the loading of SMCNs ranging from 0.1 to 0.25 g, which is equal to catalyst-to-fructose (C:F) ratio of 0.083 to 0.208, was conducted with the reaction time and temperature of 30 min and 180 °C, respectively. As depicted in Fig. 4, C:F ratio of 0.083 resulted in 68% conversion of fructose and 42% yield of 5-HMF. An increase in C:F ratio to 0.125 and 0.167 led to enhancement in both fructose conversion and 5-HMF yield. However, a slight increase in LA yield was noticeable. Therefore, additional loading of the SMCN catalyst would contribute to conversion of fructose and promote the rehydration of 5-HMF into LA and furfural. Qi et al. reported that the presence of abundant acid sites on the solid acid catalyst would inevitably induce water molecules to involve with rehydration of 5-HMF [3]. However, the fraction of the resultant 5-HMF was still predominate with the presence of SMCN catalyst.

3.2.4. Recyclability of SMCNs catalyst

Recyclability of the SMCN catalyst was also evaluated with experimental conditions of 180 °C, 30 min and C:F ratio of 0.167. It is noteworthy that the spent SMCNs could be separated from the resulting liquid product after reaction by magnetic decantation using an external magnet, as shown in Fig. S3. Spent SMCNs were then regenerated by washing several times with DI-water and ethanol, followed by drying at 70 °C in an electrical oven. Interestingly, a high fructose conversion and 5-HMF yield of 81% and 54%, respectively, could still be maintained after three cycles, as exhibited in Fig. 5. Because recycling of

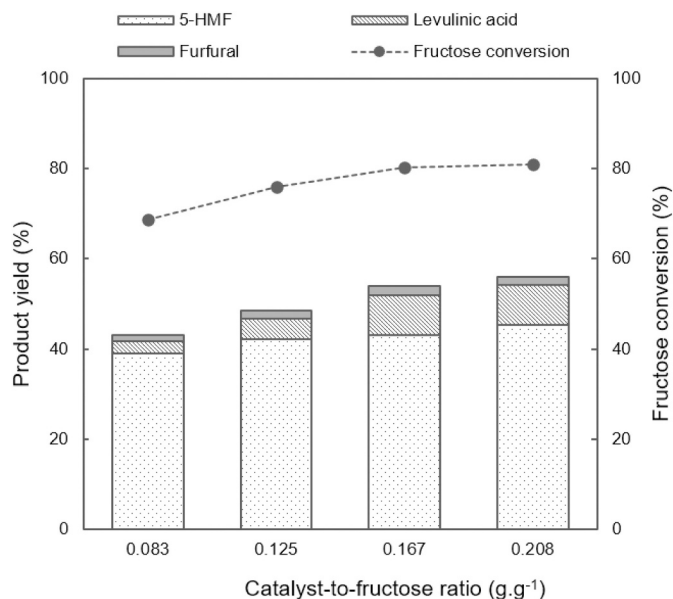


Fig. 4. Effect of catalyst-to-fructose ratio on fructose conversion and product yield (Conditions: 1.2 g fructose, 30 mL DI-water, 30 min, 180 °C).

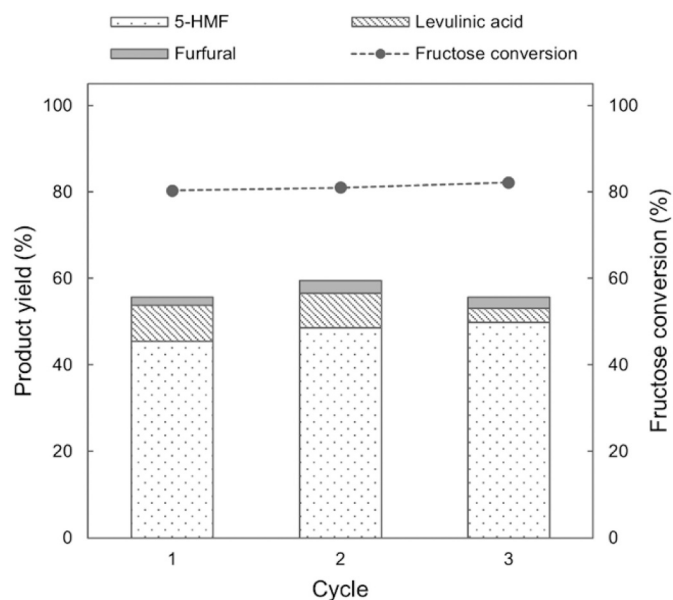


Fig. 5. Fructose conversion and product yield in recycling runs using regenerated SMCNs (Conditions: 1.2 g fructose, 0.2 g SMCN, 30 mL DI-water, 30 min).

heterogeneous catalyst is mandatory for its practical use [30,31], these reusability results reveal the potential of SMCNs as a green and sustainable catalyst for the fructose conversion in aqueous reaction system.

4. Conclusions

Magnetic carbon nanoparticles were successfully synthesized from eucalyptus oil and functionalized with sulfonic group to be employed as a sustainable catalyst for dehydration of fructose to 5-HMF. Reaction time, temperature and SMCN catalyst loading were controlled to optimize the formation of 5-HMF while LA, furfural, humin were also analyzed. The highest yield of 5-HMF (51.6%) could be achieved with reaction time of 30 min at 180 °C and C:F ratio of 0.167. Typical SMCN catalysts could be employed in fructose conversion up to three cycles without a significant decrease in catalytic performance. The environmentally benign preparation of SMCNs from renewable carbon feedstock with its notable reusability could lead to an eco-friendly production of platform chemicals.

Declaration of Competing Interest

None.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.catcom.2020.106229>.

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