



Catalytic Conversion of Glucose into 5-hydroxymethyl furfural over Spent Dry Batteries as Catalysts

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Abstract

Biomass is a popular renewable carbon source because it has a lot of potential as a substitute for scarce fossil fuels and has been used to make essential compounds like 5-hydroxymethylfurfural (HMF). One of the main components of biomass, glucose, has been extensively studied as a precursor for the production of HMF. Several efforts have been made to find efficient and repeatable procedures for the synthesis of HMF, a chemical platform used in the manufacturing of fuels and other high-value compounds. Sulfonated graphite (SG) was produced from spent dry batteries and utilized as a catalyst to convert glucose to 5-hydroxymethylfurfural (HMF). Temperature, reaction time, and catalyst loading were the variables studied. When dimethyl sulfoxide was utilized as the solvent at 180°C after 3 hours of reaction time, the greatest HMF yield, glucose conversion, and selectivity were attained, with 56.53 %, 97.5 %, and 57.979 %, respectively. This study demonstrates how to manufacture sulfonated graphite with increased catalytic activity for converting glucose to key biobased platform chemicals in a long-term sustainable manner.

Keywords: Biomass, 5-hydroxymethylfurfural (HMF), glucose conversion.

Received on 03/02/2022, Accepted on 13/03/2022, published on 30/03/2022

<https://doi.org/10.31699/IJCPE.2022.1.4>

1- Introduction

Without a doubt, one of civilization's most basic requirements, energy, must be readily available. Energy underpins pillars of modern society such as technical advancement, scientific achievement, and cultural advancement.

Conventional fossil fuels such as petroleum, coal, and natural gas are currently the primary sources of energy and chemicals. Fossil fuel is made up of organic materials that have been chemically changed over thousands of years by extreme heat and pressure. Petroleum is used to make nearly 95 % of chemicals, in addition to being a source of energy and fuel [1].

As the world's population grows and living standards rise, the need for energy and chemicals rises as well. However, fossil fuels, such as petroleum, have a finite supply and will not be able to provide the world's energy needs indefinitely [2]. Instead of refining petroleum to generate hydrocarbons derivatives like liberates, fuels, and chemicals, we may refine renewable (wood, municipal and industrial wastewater, agriculture, and paper wastes) into fuel.

Sugars are raw materials for a variety of products, including bioplastics, ethanol, citric acid, and other compounds. The need for energy has surged in recent years, resulting in increased oil prices. As costs rise, biomass-derived products become more cost-effective and environmentally friendly than fossil-fuel-derived products [3].

As a result, alternative energy, fuel, and chemical production sources and technologies must be developed to at least supplement current petroleum-based sources. Biomass offers a possible alternative to petroleum as a renewable energy source. Organic materials generated from live or recently living creatures are classified as biomass. Solar, wind, geothermal, nuclear, and hydropower are examples of renewable alternative energy sources. Even though none of the above resources can cover all of our future energy needs, the potential contribution of lignocellulosic biomass is enormous due to its widespread availability [4]. The annual global biomass production is predicted to be 170 billion metric tons. Furthermore, biomass plays an important part in carbon balancing since CO₂ produced during usage can be offset by CO₂ fixation by plants during photosynthesis [5].

Among the several methods for valorizing lignocellulosic biomass, catalytic dehydration of glucose to create 5-hydroxymethylfurfural (HMF) is a promising one. HMF (5-hydroxymethyl-2-furaldehyde, 5-hydroxymethyl-2-furancarboxaldehyde) is a frequent chemical produced by heat treatment of carbohydrate-containing foods in the presence of amino acids [6].

Dried fruits, coffees, cereals, and baking items all contain it naturally. 5-hydroxymethylfurfural (HMF), a five-member aromatic ring molecule with hydroxymethyl and aldehydefunctional groups, is widely used in the synthesis of medicines, polymers, fine chemicals, and other organic derivatives.

HMF is made by dehydrating monosaccharides, disaccharides, and polysaccharides with an acid catalyst [7]. Acid-catalyzed dehydration of C6 carbohydrates, such as fructose and glucose, is the preferred method for obtaining HMF. Due to their availability and abundance in agricultural side streams and other wastes, glucose-based polymers, particularly those generated from lignocellulosic sources, are preferred feedstocks.

The development of catalytic methods that can efficiently combine the complicated multi-step transformation processes involved in using biorenewable feedstock for the sustainable synthesis of HMF is of special interest [8].

HMF is a platform chemical made by removing triple water molecules from C6 carbohydrate in the presence of an acid catalyst [9]. The impact of acid treatments such as HCL, HF, and HNO₃ on the characteristics of a supported AC catalyst was discovered that acid treatment greatly affected the surface chemical characteristics of the AC and increased its surface area.

It was thought that HNO₃ may oxidize certain complexes and produce acidic groups. Surface acidity rises, resulting in a more homogeneous distribution on AC [10]. Metal chlorides [11], mineral acids [12], carbon solid acids [13], ion exchange resin [14], zeolites [15], solid metal phosphates [16] and sulfated zirconias [17] have all been used as heterogeneous or homogeneous acid catalysts in this reaction.

Heterogeneous catalysts, in comparison to dissolving homogeneous acid catalysts, have various advantages, including the ability to be simply separated or recycled and the lack of equipment corrosion. Solid acid catalysts also enable surface acidity adjustment and operation in harsh reaction conditions. For diverse carbohydrate conversions, various kinds of acid sites such as Bronsted acid, Lewis acid, and bifunctional acid were added onto solid catalyst [18].

Using Bronsted acid catalysts, fructose can be transformed to HMF in a step process. Because the reaction is frequently a two-step process requiring the catalysts to have two types of active sites, the conversion of glucose to HMF is substantially more complex than the transformation of fructose to HMF [9].

Catalytic reaction glucose isomerization to fructose in the presence of Lewis acids, enzymes, or bases is the initial step. The second stage of fructose dehydration to HMF is driven by Bronsted acids. Fig. 1 shows the glucose conversion to 5-hydroxymethylfurfural.

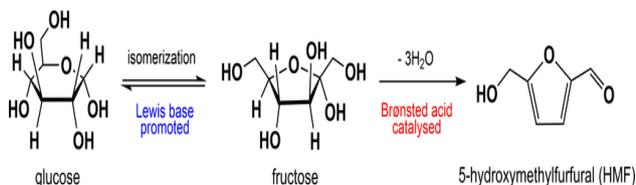


Fig. 1. Glucose conversion to 5-hydroxymethylfurfural (HMF)

Recent research reveals that under sufficient Bronsted acidity, glucose can also be directly dehydrated to HMF, bypassing the glucose-fructose isomerization [19]. Some solid acids treated with Lewis acid sites or Bronsted acid, on the other hand, release only small amounts of HMF [18]. Strong Bronsted acids produce significant HMF yields, but they also cause unwanted HMF degradation to humins [20] and levulinic acid [21]. Dimethylsulfoxide (DMSO) [22], water [23], tetrahydrofuran (THF) [17], methyl isobutyl ketone (MIBK) [24], ionic liquids (ILs) [25] and γ -valerolactone (GVL) [26], have all been studied as solvents to decrease the development of potential byproducts.

Many domestic items, such as wireless mice, digital cameras, toys, flashlights, clocks, and so on, use alkaline batteries as power sources [27]. Aside from the batteries gathered from customers, factory-produced wasted batteries are a significant waste source that must not be overlooked. Because each factory's annual production volume is typically over a hundred million pieces, the quantities of co-produced wasted batteries in the manufacturing lines are just too vast to be ignored [28].

Alkaline batteries account for 68% of all battery sales in Switzerland [29], 60% in the UK [30], and 47% in the EU [31], hence recycling spent batteries is critical for environmental safety, human health, and resource efficiency [27].

This work aims to convert glucose into 5-hydroxymethyl furfural, preparation of catalyst from used dry batteries, and study the parameters that are effective on the glucose conversion, HMF yield and selectivity, the studied parameter are: temperature, time and catalytic loading were examined to get the optimum results.

2- Experimental Work

2.1. Materials

Without additional purification, all of the reagents were employed. Dry batteries, D-glucose (99.5%), was provided by HiMedia Laboratories Pvt. Ltd. (Mumbai, India), 5-hydroxymethyl-2-furaldehyde (HMF, 98%, Shanghai Macklin Biochemical Co., Ltd), sulfuric acid (H₂SO₄, 98 wt%), dimethylsulphoxide (DMSO, Mumbai, 99%), hydrochloric acid (HCl, 36.5%), Glycerine bath, throughout the studies, deionized water was used.

2.2. Catalysts Preparation

The catalyst preparation procedure included two steps: graphite preparation and sulfonation of the graphite obtained. Utilized dry batteries were used to make the graphite. We began by gathering the spent batteries and separating the cathode electrode (graphite rod) from the anode electrode.

The cathode electrode (graphite rod) was next grounded by a high-speed rotating having to cut machine, and finally through mesh sieve to yield tests to the desired particle size, before being rinsed with distilled water at ambient temperature and dried for 2 hours at 100 °C.

Sulfonation of graphite: 5 g of graphite samples (to get 5 grams of graphite, we need about 5 batteries because the weight of the graphite electrode for each battery is about =1.03) were placed in a three-necked round bottomed flask with a concentrated sulfuric acid solution (50 ml), the flask has been heated to 160 °C for 5 hours by a glycerine bath with magnetic stirring under reflux, as shown in Fig. 2.

The solids fraction were recovered by filtration and cleaned with deionized water to eliminate any impurities from the solid material after preserving a sulfonation reaction. The sulfonated samples were cleaned with distilled water many times until the pH of the filtrate was normal, then dried at 100°C for 2 hours. The resulting solid was labeled SG and stored in a desiccator until it was needed.

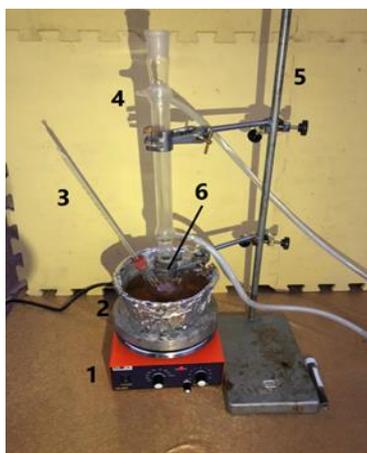


Fig. 2. Photograph of the experimental system (1)magnetic stirrer (2)glycerine bath (3)thermometer (4)condenser (5)stand (6) three-necked round bottomed flask

2.3. Characterization of Catalyst

Surface area and Pore volume measurements, Brunauer Emmett- Teller (BET) and pore volume, according to 9277- 2010 iso method was performed to measure the surface area and pore volume, country USA, Thermo Finnigan, device range (0.1- 2000 m²/g), at Petroleum R and D Center (PRDC) Laboratory, Ministry of oil/ Iraq.

2.4. Conversion of Glucose to HMF

5 grams of glucose were combined with 50 ml DMSO in a 100 ml three-necked round bottomed flask, then the appropriate amount of SG was added as a catalyst. After that, the reactor was immersed in a glycerine bath.

The reaction conditions were as follows: The magnetic stirrer speed was 550 rpm, the temperature range was 120 to 200 °C, the time was 0.5 to 4 hours, the catalyst loading was 0.025 to 2.5 grams, and DMSO as a solvent was utilized.

After the reactions, the reactor was immersed in cold water to cool to room temperature, and the liquid phases were removed using filtering.

2.5. Product Analysis

The amount of HMF and glucose in the reaction solutions was determined using high-performance liquid chromatography (HPLC) as analytical method. A separation module LC-2030, a column ODS (250 × 4.6) mm, and a detector type PDA were used in the HPLC study (photodiode array detector). The mobile phase for the Shimadzu (3D) type of HPLC was 80% methanol and 20% H₂O; flow rate was 1 ml/min; volume was 20 μl; particle size was 5 μm). The UV detection for HMF was performed at 285 nm, with the column temperature kept constant at room temperature. The concentrations of substrate and product were obtained using a standard calibration curve, and the glucose conversion, HMF yield, and HMF Selectivity were computed using the equations below:

$$\text{Conversion (\%)} = \frac{\text{mole of starting glucose} - \text{mole of glucose remaining}}{\text{mole of starting fructose}} \times 100 \quad (1)$$

$$\text{HMF Yield (\%)} = \frac{\text{moles of HMF produced}}{\text{mole of starting glucose}} \times 100 \quad (2)$$

$$\text{HMF Selectivity (\%)} = \frac{\text{moles of HMF produced}}{\text{mole of starting glucose} - \text{mole of glucose remaining}} \times 100 \quad (3)$$

3- Results and Discussion

3.1. Catalyst Characterization

The results of BET analysis for graphite and sulfonated graphite are reported in Table 1. An approximately large change was observed in the surface area of sulfonated graphite as compared to graphite. Maximum pore volume (0.0449 cm³/g) was observed for sulfonated graphite as compared to graphite 0.0022 cm³/g showing an increase in the porous structure of the catalyst. Hence, chemical modification has a significant role to increase the surface area of the catalyst.

Table 1. Physical structure characterization of graphite and sulfonated graphite

Properties	Graphite	Sulfonated graphite
Total surface area (B.E.T) m ² /g	0.579	79.972
Total pore volume, cm ³ /g	0.0022	0.0449

3.2. Effects Of Operating Parameters

a. Temperature effect on glucose dehydration

The reaction temperature has a significant impact on the yield of 5-HMF formed from glucose using sulfonated graphite (SG) as a catalyst. As shown in Fig. 3, when the reaction temperature was raised from 120 to 180 °C, the peak 5-HMF yield increased from 26.4% to 46.25%, while glucose conversion and selectivity increased (64.8–87.5% and 40.74–52.85%, respectively), indicating that the higher temperature resulted in a faster peak value and higher HMF yield, when the temperature hit 200°C, the yield of HMF reduced by 43.84 %. Mostly because of unexplained soluble polymers and humins that resulted from side reactions.

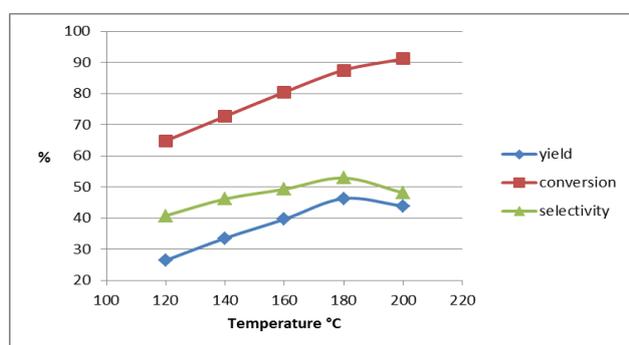


Fig. 3. The influence of temperature on glucose dehydration to 5-HMF in DMSO over SG catalyst ($m_{\text{cata}} = 1\text{g}$, time = 2 hr)

b. Time Effect on Glucose Dehydration

The results of the influence of time on the dehydration reaction of glucose are shown in Fig. 4. The yield of HMF increased dramatically with increasing reaction time, rising from 23.3% to 51.76% when the reaction period was increased from 0.5 to 3 hours. The HMF yield was then reduced to 44.18% after a four-hour dip. As reported previously [10], the lower HMF production after 4 hr could be due to additional converting to by-products. As a result, the reaction using sulfonated graphite (SG) as a catalyst was chosen to be examined at 160°C for 3 hours.

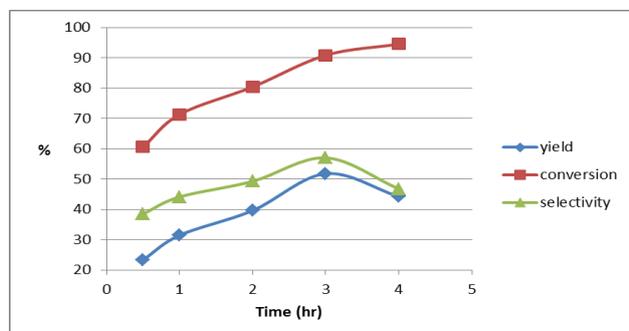


Fig. 4. The influence of time on glucose dehydration to 5-HMF in DMSO over SG catalyst ($m_{\text{cata}}=1\text{g}$, temperature = 160°C)

c. Catalyst Loading Effect on Glucose Dehydration

The effect of varying catalyst amounts in the range of 0.25 to 2.5 g was investigated. The best yield of HMF (53.2%) was obtained with a catalyst loading of 2 g, according to the results given in Fig. 5. The HMF yield fell when the amount of catalyst was increased; for example when the catalyst loading was 2.5 g, the HMF yield dropped to 51.8 %, likely because the system's high acidity was susceptible to the development of side-reactions.

At 160°C for 2 hours, the effect of H_2SO_4 loading on the dehydration of glucose to HMF with DMSO as the solvent. Due to the additional conversion of HMF to LA, increasing the H_2SO_4 level resulted in a drop in HMF production.

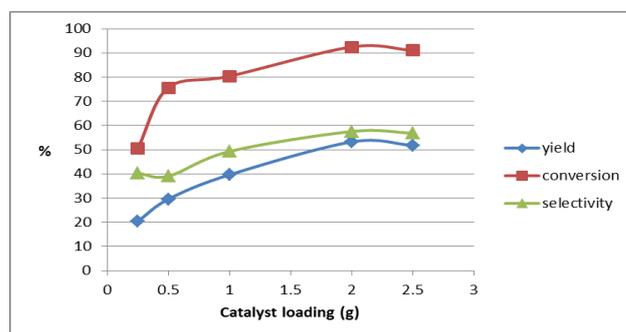


Fig. 5. The influence of catalyst loading on the glucose dehydration to 5-HMF in DMSO over SG catalyst (time = 2hr, temperature = 160°C)

d. Optimum Operating Condition

All of the experiments were studied, and the best operating parameters for dealing were established based on temperature, time, and catalyst loading. The best conditions for producing 5-HMF from glucose were found to be at 180°C for 3 hr, with a catalyst loading of 2g and DMSO as the solvent. The glucose conversion rate was 97.5%, and the yield of HMF was 56.53%. For these conditions Fig. 6 shows dehydration of glucose to 5-HMF.

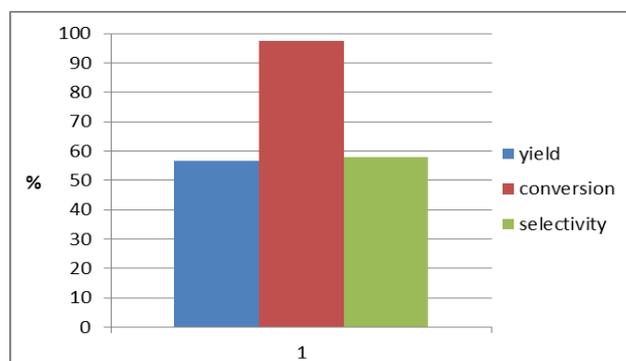


Fig. 6. The glucose dehydration to 5-HMF in DMSO over SG catalyst (time = 3hr, temperature = 180°C, $m_{\text{cat}} = 2\text{g}$)

4- Conclusions

This research proposes a more environment friendly and intensive approach for effective conversion of biomass into valuable products. Both catalysts and solvents are required for the conversion of glucose to 5-HMF. The use of sulfonated graphite (SG) as a catalyst, which is generated from spent dry batteries, has been shown to be promising method, yielding a maximum of 56.53 % of 5-HMF at 180°C for 3 hours with a catalyst loading of 2g and DMSO as the solvent. The DMSO solvent helps to increase product yield while also shortening reaction times. This technique of converting glucose to 5-HMF is environment friendly and energy efficient.

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التحويل التحفيزي للجلوكوز إلى 5-هيدروكسي ميثيل فيرفرال فوق البطاريات الجافة المستعملة كعامل مساعد

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الخلاصة

تعتبر الكتلة الحيوية مصدرًا شائعًا للكربون المتجدد لأنها تتمتع بإمكانيات كبيرة كبديل للوقود الأحفوري الشحيح ، وقد تم استخدامها لصنع مركبات أساسية مثل 5-هيدروكسي ميثيل فيرفرال (HMF). تمت دراسة الجلوكوز ، وهو أحد المكونات الرئيسية للكتلة الحيوية ، على نطاق واسع باعتباره مقدمة لإنتاج HMF. تم بذل العديد من الجهود لإيجاد إجراءات فعالة وقابلة للتكرار لتركيب HMF ، وهي منصة كيميائية تستخدم في تصنيع الوقود والمركبات الأخرى عالية القيمة. تم تصنيع الكرافيت المسلفن (SG) من البطاريات الجافة المستهلكة واستخدامه كعامل مساعد لتحويل الجلوكوز إلى 5-هيدروكسي ميثيل فيرفرال (HMF). كانت درجة الحرارة ووقت التفاعل وتحميل العامل المساعد من بين المتغيرات التي تمت دراستها. عندما تم استخدام ثنائي ميثيل سلفوكسيد كمذيب عند 180 درجة مئوية بعد 3 ساعات من وقت التفاعل ، تم تحقيق أكبر ناتج HMF وتحويل الجلوكوز والانتقائية بنسبة 56.53% و 97.5% و 57.979% على التوالي. توضح هذه الدراسة كيفية تصنيع الجرافيت المسلفن مع زيادة نشاط العامل المساعد لتحويل الجلوكوز إلى مواد كيميائية أساسية قائمة على أساس حيوي بطريقة مستدامة طويلة الأجل.

الكلمات الدالة: الكتلة الحيوية ، 5-هيدروكسي ميثيل فيرفرال (HMF) ، تحويل الجلوكوز .