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Data on the synthesis and characterizations of carboxylated carbon-based catalyst from eucalyptus as efficient and reusable catalysts for hydrolysis of eucalyptus

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ARTICLE INFO

Article history:

Received 16 March 2020

Revised 21 March 2020

Accepted 25 March 2020

Available online 17 April 2020

Keywords:

Eucalyptus

Biomass

Biofuel

Carboxylated carbon catalyst

Heterogeneous catalysts

ABSTRACT

The presented article reports the preparation and characterization of heterogeneous carbon catalyst enriched with carboxylic group denoted as (ECS) from *Eucalyptus* as an efficient catalyst for the hydrolysis of woody *Eucalyptus* biomass. The fabrication process is based on the ball milling of *Eucalyptus* as a carbon source in the presence of dry ice as an oxidizing agent followed by acidification with the assistance of hydrochloric acid. The data are including the schematic for the full synthesis steps and characterization tools in addition to the thermogravimetric analysis and proton nuclear magnetic resonance analysis for the ECS catalyst. Meanwhile, the catalytic performance of ECS catalyst towards the hydrolysis of *Eucalyptus* was measured under different temperatures ranged from 160 to 200 °C. The ECS catalyst allowed the selective hydrolysis of *Eucalyptus* to glucose and xylose, as proved by high-performance liquid chromatography. The

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<https://doi.org/10.1016/j.dib.2020.105520>

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data herein are associated with the article entitled “ Unveiling one-pot fabrication of scalable and reusable carboxylated heterogeneous carbon-based catalyst from *Eucalyptus* plant with the assistance of dry Ice for selective hydrolysis of *Eucalyptus* Biomass” [1].

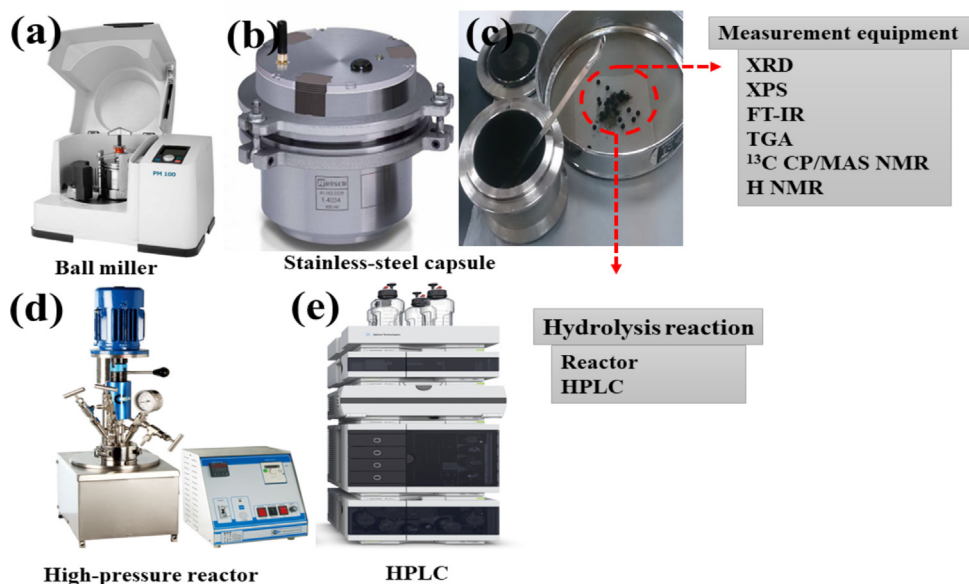
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Specifications table

Subject	Chemistry
Specific subject area	General chemistry, including developing catalysis for hydrolysis of <i>Eucalyptus</i> to xylose and glucose fuel.
Type of data	Image Chart Graph Figure
How data were acquired	Thermogravimetric analysis (TGA) was recorded on (TGA, STA 449 F3, NETZSC, Germany). Proton nuclear magnetic resonance (^1H NMR) spectra were measured by BrukerAvance III 400 MHz spectrometer equipped with a 5 mm SEI probe with a z-gradient (Bruker, Rheinstetten, Germany). The high-performance liquid chromatography ((HPLC), Agilent 1260 series, USA) with an Aminex HPX-87H column (300 × 7.8 mm, Bio-Rad, USA). Photographs for the Ball miller system were Canon DSLR Camera EOS 80D 18 mm Lens and collected using Microsoft PowerPoint. All the charts for TGA, HPLC, and ^1H NMR were drawn using OriginPro 2018.
Data format	Raw data TGA, HPLC, and ^1H NMR
Parameters for data collection	The hydrolysis process was carried out under different temperatures, including 160 °C, 180 °C, and 200 °C.
Description of data collection	The preparation steps, analysis apertures, thermal stability, and chemical structure of the as-obtained catalysts. The hydrolysis of <i>Eucalyptus</i> biomass was carried out at different reaction temperatures along with the identification of the products of the hydrolysis using HPLC.
Data source location	Key Laboratory of Bioelectrochemistry & Environmental Analysis of Gansu Province, College of Chemistry & Chemical Engineering, Northwest Normal University, Lanzhou 730070, P. R. China Center for advanced materials, Qatar University, Doha 2713, Qatar Tianjin Key Laboratory of Molecular Optoelectronic Science, Department of Chemistry, School of Science, Tianjin University, Tianjin, 300072, P. R. China
Data accessibility	The data are provided herein in this article. All the raw data were provided as a supplementary Microsoft Excel file contains the raw data for TGA, ^1H NMR, and hydrolysis process.
Related research article	Abdu, H.I., et al. Unveiling one-pot fabrication of scalable and reusable carboxylated heterogeneous carbon-based catalyst from <i>Eucalyptus</i> plant with the assistance of dry ice for selective hydrolysis of <i>Eucalyptus</i> Biomass. [1] https://doi.org/10.1016/j.renene.2020.02.034

Value of the data

- Controlling the preparation and characterizations of heterogeneous carbon-based catalysts is vital in the biofuel application.
- The carboxylated carbon catalyst was prepared from *Eucalyptus* and used for the hydrolysis of woody *Eucalyptus* biomass.
- The catalytic hydrolysis *Eucalyptus* was benchmarked at different temperatures to understand the reaction kinetics.



Scheme 1. Illustrates the detailed schematic of the experimental apparatus and measurement equipment.

- The as-obtained catalyst can be easily recycled and reused after the hydrolysis process.
- The presented data are essential for the preparation of reusable heterogeneous catalyst from various plants.

1. Data description

The data presented in this article are related to the rational design of efficient and reusable ECS catalysts from *Eucalyptus* plant and its utilization for the hydrolysis of *Eucalyptus* plant [1]. The data including the detailed schematic of the experimental, characterization, and hydrolysis application apparatus, including ball-miller for the preparation of ECS catalyst from *Eucalyptus*, high-pressure reactor for measuring the catalytic performance over *Eucalyptus* plant, and HPLC for determination the hydrolysis products as well as characterization tools (Scheme 1). The photographs for showing the difference between the pristine *Eucalyptus* plant and the ECS catalyst (Fig. 1). This is in addition to using the ¹H NMR for identifying the chemical structure of the as-obtained ECS catalysts compared to natural *Eucalyptus* plant (Fig. 2) and the TGA to investigate the thermal stability of ECS catalysts obtained at different milling times including 24, 36, 45, and 64 h (Fig. 3). Then, the hydrolysis products of *Eucalyptus* on the as-synthesized ECS catalyst carried out under different temperatures, including 160 (Fig. 4a), 180 (Fig. 4b) and 200 °C (Fig. 4a) as well as the hydrolysis products obtained during the reaction times (Fig. 4).

2. Experimental design, materials, and methods

2.1. Preparation process of ECS

Scheme S1 shows a schematic of the experimental and characterization apparatus for the formation of ECS catalyst from woody *Eucalyptus* biomass [1,2]. The preparation process, including mixing *Eucalyptus* and dry ice in a stainless-steel pot (250 ml) contains stainless-steel balls

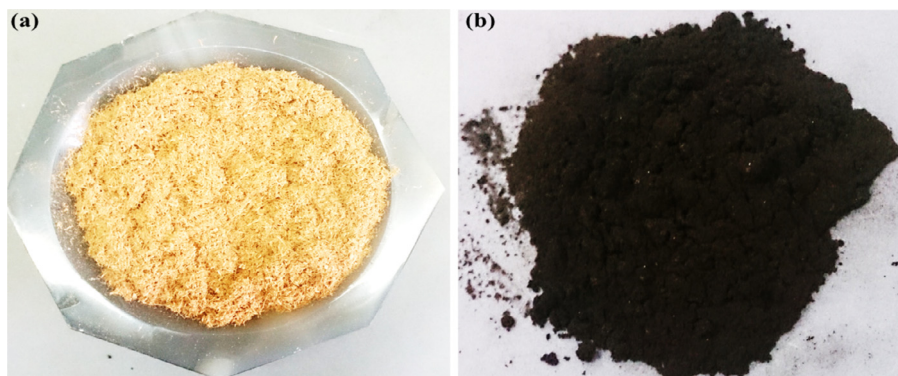


Fig. 1. Photographs of (a) *Eucalyptus* and (b) ECS catalyst.

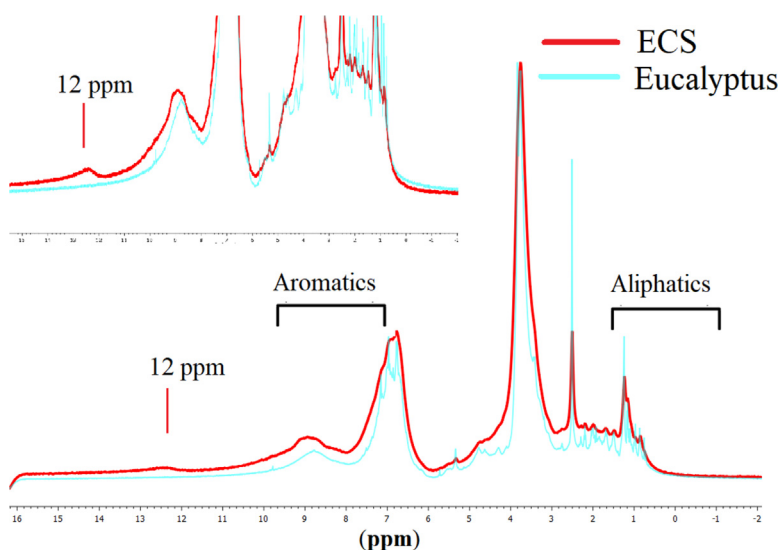


Fig. 2. The ¹H NMR spectrum of ECS and pristine *Eucalyptus*. The insight shows the magnification of the area between 16 and 7 ppm for both ECS catalyst and *Eucalyptus* to confirm the formation of COOH group inside ECS catalyst.

(5 mm), and then milling at 550 rpm using Ball miller (Fritsch P-6 planetary) for 2 h, followed by filtration, washing, and finally drying under air. The content of the carboxylic (COOH) group inside ECS catalyst could be easily controlled by adjusting the milling time. The obtained ECS catalysts were confirmed using various characterization tools such as TGA for thermal stabilities and ¹H NMR for chemical structure [1,3,4]. The high-pressure reactor for the hydrolysis process and HPLC for the identification of the products.

Fig. 1 shows the photographs of pure *Eucalyptus* and the obtained ECS, which indicates changing the color of pure *Eucalyptus* from the pale brown to deep black color after the carboxylation process and annealing (Fig. S1).

Fig. 2 shows the ¹H NMR spectra for both ECS catalyst and pure *Eucalyptus*, which both showed the signals attributed to the H of the aliphatic carbons between 0.9 and 1.6 ppm in addition to the signals between 6.3 and 9.5 ppm [5–7]. Meanwhile, the ECS catalyst reveals the signal assigned to H bonded to the C=O group at 12.0 ppm, indicating the successful formation of ECS catalyst composed of C-aromatic skeletal enriched with COOH group and some aliphatic

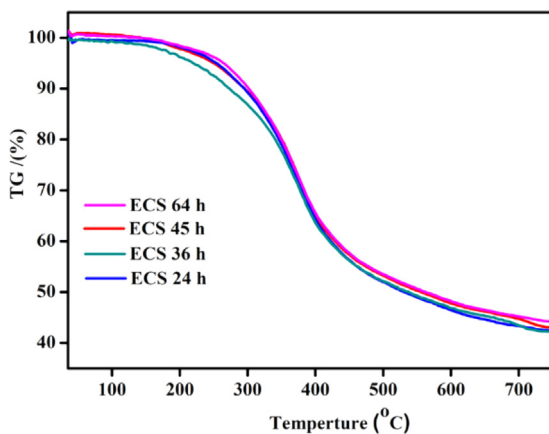


Fig. 3. The TGA for ECS catalysts prepared under different milling times.

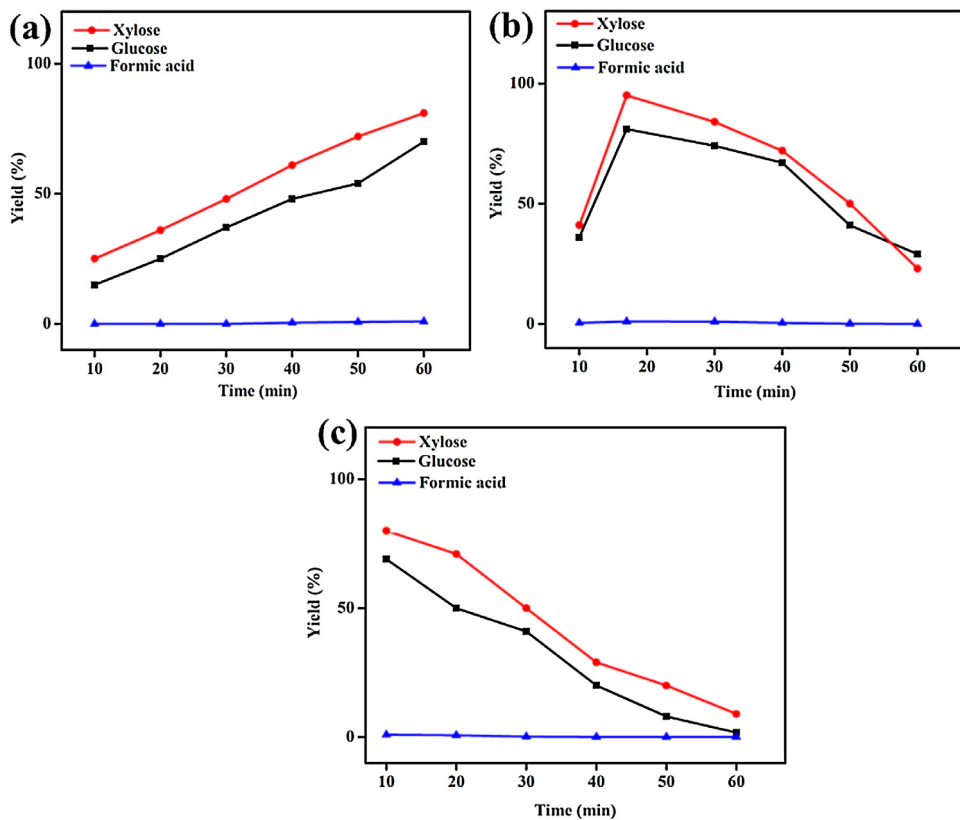


Fig. 4. Hydrolysis of *Eucalyptus* by ECS catalysts under different temperatures and times for (a) at 160, (b) 180 and (c) 200 °C.

moieties [5–7]. The ^1H NMR displayed the absence of undesired products, except the signal at 2.5 and 3.35 ppm originated from H of *dimethyl sulfoxide* solvent.

The thermal stability of the as-synthesized ECS catalyst prepared under different milling times, comprising 24, 36, 45, 64 h, were investigated by the TGA, as shown in Fig. 3. There is no any kind of noticed significant changes among all prepared ECS catalysts [8]. Meanwhile, all ECS catalysts were thermally stable until 200 °C (Fig. S3). The major decompositions of ECS were achieved between 250 and 450 °C as the carbon-based catalysts are highly stable thermally and chemically [9].

Fig. 4 depicts the catalytic performance of ECS catalyst over *Eucalyptus* benchmarked at 160, 180, and 200 °C in the presence of 120 ppm of HCl, and the reaction products were estimated using the HPLC at 10 min intervals each. It should be noticed that, before any hydrolysis process, both *Eucalyptus* and ECS were milled together for 2 h to enhance the contact between them in line with elsewhere reports [10–14]. In particular, at 160 °C, the hydrolysis of *Eucalyptus* increased with time till achieving the maximum glucose yield of (70%) and xylose yield of (82%) after 1 h (Fig. 4a). Likewise, at 180 °C, the hydrolysis activity increased quickly until 17 min to reach the maximum production yield of (81%) for glucose and (95%) of xylose, followed by quickly decreasing until reaching the lowest yield of nearly (10%) for xylose and (15%) for glucose after 1 h (Fig. 4b). At 200 °C, the hydrolysis enhanced promptly to reach the maximum value of glucose (69%), and xylose (80%) after 10 min and then decreased quickly (Fig. 4c). Under all the temperatures used, only an inferior amount of furfural acid was detected. All data showed that the kinetics of hydrolysis at 200 °C was higher than that at 180 °C and 160 °C, respectively [15]. Meanwhile, the optimum reaction temperature was 180 °C.

Acknowledgments

This work was supported by the [Natural Science Foundation of China](#) (Grant Nos. 21575115, 21327005); Program for Chang Jiang Scholars and Innovative Research Team, Ministry of Education, China (Grant No. IRT-16R61, 2017D-01); the Program of Innovation and Entrepreneurial for Talent, Lanzhou, Gansu Province, China (Grant No. 2014-RC-39). This publication was supported by Qatar University Internal Grant No. Project QUCC-CAM-20/21-2. The findings achieved herein are solely the responsibility of the authors.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships, which have, or could be perceived to have, influenced the work reported in this article.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:[10.1016/j.dib.2020.105520](https://doi.org/10.1016/j.dib.2020.105520).

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