Research Square

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Green Valorization of Corn Cob Waste via a Novel HCOOH–NaOH Dehydrogenation System for High-Yield 5-Hydroxymethylfurfural Production

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Abstract

A 5-hydroxy methyl furfural (5-HMF) is contemplated a key bio-based principles chemical that presents potential in important substrate of various bio-base polymer materials. A novel system of corn cob dehydrogenated with methanoic acid (HCOOH): sodium hydroxide base (NaOH) has been studied. The dehydrogenation process is suggested with HCOOH:NaOH utilizing of method 1 at 120°C for 180 min obtained a maximum 5-Hydroxy Methyl Furfural yield (5-HMF yield) of 80.17 ± 0.11% when compared with 18.23 ± 0.05 % 5-HMF yield of method 2 and 8.92 ± 0.07 % of method 3, significantly. Interestingly, the% 5-HMF yield of humin can be enhanced by corn cob dehydrogenated with HCOOH:NaOH at 120°C for 180 min all of methods. By considering the fourier transform infrared spectrometer analysis (FTIR analysis) of humin structure, the 5-HMF structure of corn cob dehydrogenated with HCOOH:NaOH at 120°C for 180 min of method 1 can be appeared while the humin structure method 2 and 3 of HCOOH:NaOH dehydrogenation at 120°C for 180 min cannot be appeared, indicating that the high performance 5-HMF product can be activated with novel technique of green chemistry reaction through method 1 reaction. Furthermore, These green chemistry reaction technique is first reported, demonstrating that 5-HMF of corn con dehydrogenated with HCOOH:NaOH mixture solution.

Statement of Novelty

This study introduces a previously unreported HCOOH–NaOH dehydrogenation system enabling highly selective and high-yield (80.17 ± 0.11%) production of 5-HMF from corn cob under mild conditions, without complex catalysts. FTIR evidence reveals a distinct reaction pathway unique to the optimized system. This work establishes a simplified, scalable, and sustainable platform for efficient biomass valorization toward next-generation biorefineries.

1 Introduction

Biorefinery produced with biomass that main composition (cellulose, hemicellulose and lignin) are increased value for several purposes [1–2]. Interestingly, the utilization of biomass 75% of cellulose is alternative production [3] for bio-base polymer industry. By considering the biomass for substrate of biomass increased value, such as bio-fuels and bio-base material industrial, corn bobbiomass has high cellulose quantity (≥ 30% cellulose composition) [4–5]. This cellulose can be converted to monosaccharide of glucose product to surrogate toxic chemical [6].

A 5-hydyoxy methyl furfural [5-HMF] is key bio-base (non-toxic) chemical from biomass. In fact the 5-HMF received with biomass is bio-base renewable material which is alternative to produced substrate (2,5-furan dicarboxylic acid; FDCA) for bioplastic industrial [7]. The 5-HMF is derived from hydrolysis, isomerization into fructose and dehydrogenation steps. By focusing the interested step of 5-HMF production is hydrolysis and isomerization into fructose because both of hydrolysis and isomerization into fructose steps can enhance of high performance 5-HMF substrate (glucose). Then, the glucose enhancement of corn cob added with base such as potassium hydroxide (KOH) and sodium hydroxide

(NaOH) in hydrolysis reaction for 5-HMF production can be increased at 90°C before glucose isomerized into fructose product [8]. Interestingly, the high 5-HMF yield is produced with Lewis acids isomerization of fructose dehydrogenated with Lewis acids. For the Lewis acid, the weak acid of methanoic (HCOOH) is the low toxicity. Moreover, the lignocellulose is generally denatured with $HCOO^-$, suggesting that the HCOOH is approximately used with dehydrogenation of green chemistry reactions for 5-HMF production. However, the dehydrogenation efficiency for 5-HMF production can be enhanced by the combination of acid or base solution [9–10]. Although, the performance cellulose separation of corn cob pretreatment enhanced with HCOOH:NaOH of acid-base mixture solution [11–13] has been demonstrated, the green chemistry reactions (hydrolysis, isomerization into fructose and dehydrogenation) have yet to be integrated into biorefinery process.

In this work, we present a concept biorefinery process of corn cob dehydrogenated with 100°C, 110°C, 120°C, 130°C and 140°C at 0, 60, 120, 180 and 240 min for key purpose: (i) the production of 5-HMF yield of method 1 from the cellulose of corn cob added with NaOH before HCOOH addition. (ii) the production of 5-HMF yield of method 2 from the cellulose of corn cob added with HCOOH before NaOH addition and (iii) the production of 5-HMF yield of method 3 from cellulose of corn cob added with HCOOH and NaOH (HCOOH:NaOH) mixture solution. Afterward, a high-value product biorefinery of 5-HMF of method 1, 2 and 3 utilizing innovative acid-base system of hydrolysis, isomerization and dehydrogenation are detected with high performance liquid chromatography (HPLC) analysis. Furthermore, the morphology after corn cobs dehydrogenated with HCOOH:NaOH of the best method is analyzed with fourier transform infrared spectrometer (FTIR). Lastly, the mechanism of 5-HMF production included with hydrolysis, isomerization into fructose and dehydrogenation reactions are predicted with this work.

2 Materials and methods

2.1 Biological materials

Corn cobs were harvested from local farm in Cha-am, Phetchaburi, Thailand. The corn cobs were dried and kept under ultra-low humidity storage (EDRY series SL-126CA, Taiwan) and then ground with a ball mill (T-BOTA series QM 3SP4, China) to reduce the sizing with an average diameter of less than 0.4 mm, as corncob powder.

2.2 Chemical material

Methanoic acid (HCOOH, 98% p.a.), and sodium hydroxide (NaOH, 98% AR) were purchased from Sigma-Aldrich Co., Ltd. (St. Louis, Mo, USA.). All other reagents were of analytical grade purchased from Sigma-Aldrich Co., Ltd. (St. Louis, Mo, USA.).

2.3 Green chemistry reactions of 5-HMF

The cellulose, hemicellulose and lignin in the solid recovery were detected and analyzed by using National Renewable Energy Laboratory (NREL) protocol [14–15]. before the green chemistry reactions

that contained with hydrolysis, isomerization and dehydrogenation as shown in Fig. 1a, the corn cob (1.25 g, 15% solid loading) was added with 4.25 g of 3 mol/dm^3 NaOH (hydrolysis reaction) before isomerization and dehydrogenation reactions of 4.25 g of 3 mol/dm^3 HCOOH addition at the temperature increased with hot air oven (Memmert UF 100, Germany) as method 1. The 1.25 g of corn cob was added with 3 mol/dm^3 of HCOOH (4.25 g of HCOOH) before 4.25 g of 3 mol/dm^3 NaOH addition at the increase of temperature (isomerization and dehydrogenation reactions) of method 2 as presented with Fig. 1b. The 1.25 g of corn cob was added with 8.50 g of 3 mol/dm^3 of HCOOH combined with 3 mol/dm^3 NaOH (HCOOH:NaOH) before increase of temperature (isomerization and dehydrogenation reactions) of method 3 as shown in Fig. 1c.

Afterward, the 5-HMF of method 1 to 3 is detected with HPLC (Dionex UltiMate 3000 series, Thermo Fisher Scientific), coupled to diode array detector (DAD 300) at the waveleangth 285 nm equipped with an ACE 5 C 18 column, using trisodium citrate buffer solotion (pH 2.5) as mobile phase at a flow rate of 1.0 mL/min at 30°C [16]. The percentage of 5-HMF yield is definded as follows (Eq. 1) [17]:

$$\text{5-HMF yield (\%)} = [\text{5- HMF}] / [\text{total sugars}] \times 100 \quad (1)$$

The [total sugars] is the concentration of total sugars analyzed with Dubois method and present in the biomass reaction and [5-HMF] is concentration of 5-HMF detected at the end of experiment.

2.4 Humin sample for fourier transform infrared spectrometer (FTIR) analysis

The humin sample was taken. A sticky black polymer was formed, that was dried with Freeze-drying for FTIR analysis. The FTIR spectra were used on a FTIR Single-Range spectrometer in ATR mode (Bruker, Germany). A background spectrum of air was recorded before each measurement. Each sample was detected in the range of 600 cm^{-1} to 4000 cm^{-1} with 8 scans per measurement are displayed as absorbance. The humin sample was freeze-drying for 48 h prior to analysis.

2.5 Statistical analysis

Analysis of variance (ANOVA) was selected to evaluate and identify significant factors. The significance level was used at 0.01 and 0.05 in SPSS (SPSS Statistics v21.0, IBM, Armonk, NY, USA). The simple Pearson correlation matrix was performed to detect the relationships of % HMF yield quantity for three times of data repeatability.

3 Results and Discussion

3.1 Effect of time on corn cob dehydrogenated with HCOOH:NaOH methods

The composition of corn cobs consists of 37.46 ± 0.05% cellulose, 36.32 ± 0.02% hemicellulose, 7.28 ± 0.05% soluble lignin, 11.90 ± 0.05% insoluble lignin and 7.04 ± 0.01% [18]. The temperature of 120°C is chosen as conditions for corn cob dehydrogenated with HCOOH:NaOH of methods 1 because the ≥ 120°C can produce of 5-HMF yield [19–20].

Therefore, the optimized time of corn cob dehydrogenation is used by fixed temperature at 120°C of method 1 to 3. For the Fig. 2, the 80.17 ± 0.11% to 8.92 ± 0.07% 5-HMF yield of method 1 to 3 at 120°C for 180 min are significantly higher than the 40.25 ± 0.09% to 2.78 ± 0.01% 5-HMF yield of method 1 to 3 at 120°C for 120 min. Furthermore, the % of 5-HMF yield of method 1 to 3 at 120°C for 180 min (80.17 ± 0.11% to 8.92 ± 0.07% of HMF yield) and of method 1 to 3 at 120°C for 240 min (80.22 ± 0.08% to 9.05 ± 0.02% HMF yield) are similar. It suggests that the suitable time of dehydration reaction at 120°C for 180 min is used for methods testing.

Table 1
The percentage of 5-HMF yield of method 1, method 2 and method 3 at 120°C for 180 min.

Method	% 5-HMF yield
Method 1	80.17 ± 0.11
Method 2	18.23 ± 0.05**
Method 3	8.92 ± 0.07**

n = 3 and **$p \leq 0.01$.

For the methods testing (method 1 to 3) as shown in Table 1, the 80.17 ± 0.11% 5-HMF yield of corn cob dehydrogenated with HCOOH:NaOH at 120°C for 180 min of method 1 is significantly enhanced when compared with the 18.23 ± 0.05% 5-HMF of method 2 and 2.78 ± 0.01% 5-HMF of method 3, indicating that the increase of % 5-HMF yield of addition of HCOOH of method 1 is high performance dehydrogenation. However, the optimized temperature for corn cob dehydrogenation of method 1 can be complete tested in the next section.

3.2 Effects of temperature on corn cob dehydrogenated with HCOOH:NaOH

For temperature optimized of dehydrogenation as shown on Fig. 3, the 80.17 ± 0.11% 5-HMF yields of corn cob dehydrogenation at 120°C for 180 min is significantly increased when compared with 45.02 ± 0.07%5-HMF at 110°C for 180 min of corn cob dehydrogenated with HCOOH:NaOH of method 1. While, the 80.17 ± 0.11%, 80.21 ± 0.05% and 80.25 ± 0.09%5-HMF yield of 120, 130 and 140°C for 180 min of corn cob dehydrogenated with HCOOH:NaOH of method 1 are similar. It demonstrates that the 120°C for

180 min of corn cob dehydrogenated with HCOOH:NaOH is the best condition for produced yield of 5-HMF.

Moreover, the 5-HMF of humin structure in method 1 dehydrogenation is appeared with fourier transform infrared spectrometer (FTIR) while the 5-HMF compounds peakes of method 2 and method 3 can never appeared. Therefore, the morphology of humin of method 1 is detected with FTIR analysis at the wavelength of 964 cm^{-1}, 1020 cm^{-1} and 1521 cm^{-1} [21–24].

3.3 Analysis of humin byproducts

The FTIR spectra detection is used for the prediction of humin structure of method 1 after freeze-drying. As shown in Fig. 4a, the FTIR spectra of furanic bands at 784 cm^{-1}, 964 cm^{-1}, 1020 cm^{-1} and 1521 cm^{-1} [25] analysis of humin and 5-HMF reference structures are appeared, indicating that the humin product of dehydration of method 1 remain display also in the 5-HMF product. By considering the Fig. 4b, the new spectra of humin are observed with 1612 cm^{-1} and 1705 cm^{-1} [26]. These C = C of 1612 cm^{-1} and C = O of 1705 cm^{-1} peaks [27] of method 1 demonstrate that the furanic (Heterocyclic organic compounds) of 5-HMF can be produced.

Additionally, the 2930 cm^{-1} of aldehyde C-H peaks of humin in method 1 can be found. Lastly, the 1600 cm^{-1} peaks of C-O-C of intact furanoid system can be complete appeared. It suggests that the 5-HMF product from FTIR peak characteristic for humin can fully be detected. However, the byproduct as levulinic acid during 5-HMF production can be present. Therefore, the byproduct (levulinic acid) analysis and 5-HMF mechanism are tested in the next section.

3.4 Mechanism pathway analysis

Before analysis of 5-HMF pathway prediction, the byproduct during 5-HMF production can be presented with Table 2.

Table 2
The percentage of 5-HMF and levulinic acid yield of method 1, method 2 and method 3 at 120°C for 180 min.

Method	% 5-HMF yield	% levulinic acid yield
Method 1	80.17 ± 0.11	Non-detected
Method 2	18.23 ± 0.05	Non-detected
Method 3	8.92 ± 0.07	Non-detected

n = 3 and $^{**}p \leq 0.01$.

The % levulinic acid yields cannot be detected while % yield of HMF has more than 80%yield of 5-HMF, demonstrating that the technique process of NaOH addition before 5-HMF dehydrogenation of green chemistry reaction is key process. By observation of the high percentage of 5-HMF supported with Fig. 4 result of FTIR analysis, the 5-HMF produced with 3 step of green chemistry reaction that consisted of hydrolysis, isomerization into fructose and dehydrogenation steps as displayed with the mechanism of Fig. 5.

For the Fig. 5, the cellulose is hydrolyzed with OH^- of NaOH at 90°C in the first step because the ≥ 80°C can be high performance for chemical hydrolysis reaction [28–29]. After hydrolysis step, the cellulose from corn cob of NaOH addition (method 1) is changed to glucose, suggesting that the glucose can be enhanced with cellulose hydrolyzed with base solution [30–31]. By considering the hydrolysis process, the NaOH can be reverted after cellulose hydrolyzed with NaOH as shown in Fig. 5. Additionally, before the reverted NaOH combined with fructose of dehydrogenation in Fig. 5, the glucose is isomerized and changed into fructose with increase of temperature [32–33]. For the dehydrogenation process at 120°C, the intermediates of 5-HMF can be enhanced with H^+ ion of fructose isolated with OH^- of NaOH before the electron (- ion) of fructose at the H eliminated with reverted NaOH location can be delocalized (3 rounds of delocalization process) as presented in Fig. 5. After 3 rounds of dehydrogenation step of Fig. 5, the 5-HMF and $3H_2O$ can be completely produced while the $HCOO^-$ of HCOOH can be linked with Na^+ of NaOH. It led to the HCOONa buffer solution, suggesting that the green chemistry reaction can be complete used of 5-HMF production in this work.

4 Conclusions

The optimized conditions of 5-hydroxy methyl furfural (5-HMF) production by the dehydrogenation of corn cobs mixed with HCOOH and NaOH of method 1 at 120˚C for180 min is the best condition because the 80.17 ± 0.11%5-HMF yield of method 1 is significantly higher than 18.23 ± 0.05 of method 2 and 8.92 ± 0.07%5-HMF yield of method 3, respectively. Afterward, the humin peaks involved 5-HMF morphology of FTIR analysis in the method 1 is enhanced, suggesting that the NaOH addition of hydrolysis process before isomerization into fructose and dehydrogenation is important technique for 5-HMF enhancement of green chemistry reaction. Additionally, the increase of 80.17 ± 0.11%5-HMF yield and 5-HMF compound of HCOOH:NaOH from green chemistry reaction (hydrolysis, isomerization into fructose and dehydrogenation) detected with fourier transform infrared spectrometer (FTIR) analysis can explain the first mechanism of 5-HMF production in this work.

Declarations

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Availability of data

All data analyzed during this study are included in this published article.

Author contributions

Manita Kuntapa and Nattapon Kongwattana performed the experiment and prepared the manuscript. Manita Kuntapa, Kaokanya Sudaprasert and Chakrit Tachaapaikoon helped to revise the manuscript Manita Kuntapa, Kaokanya Sudaprasert, Chakrit Tachaapaikoon Pichet Ninduangdee Damrongsak Arlai and Sumate Chomphutawach1supported and developed the idea for the study, analyzed the data, and read and approved the final manuscript.

Ethics approval and consent to participate

This article does not contain any studies involving human and animals participants performed by any of authors

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Competing interests

The authors declare no conflicts of interest.

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Figures

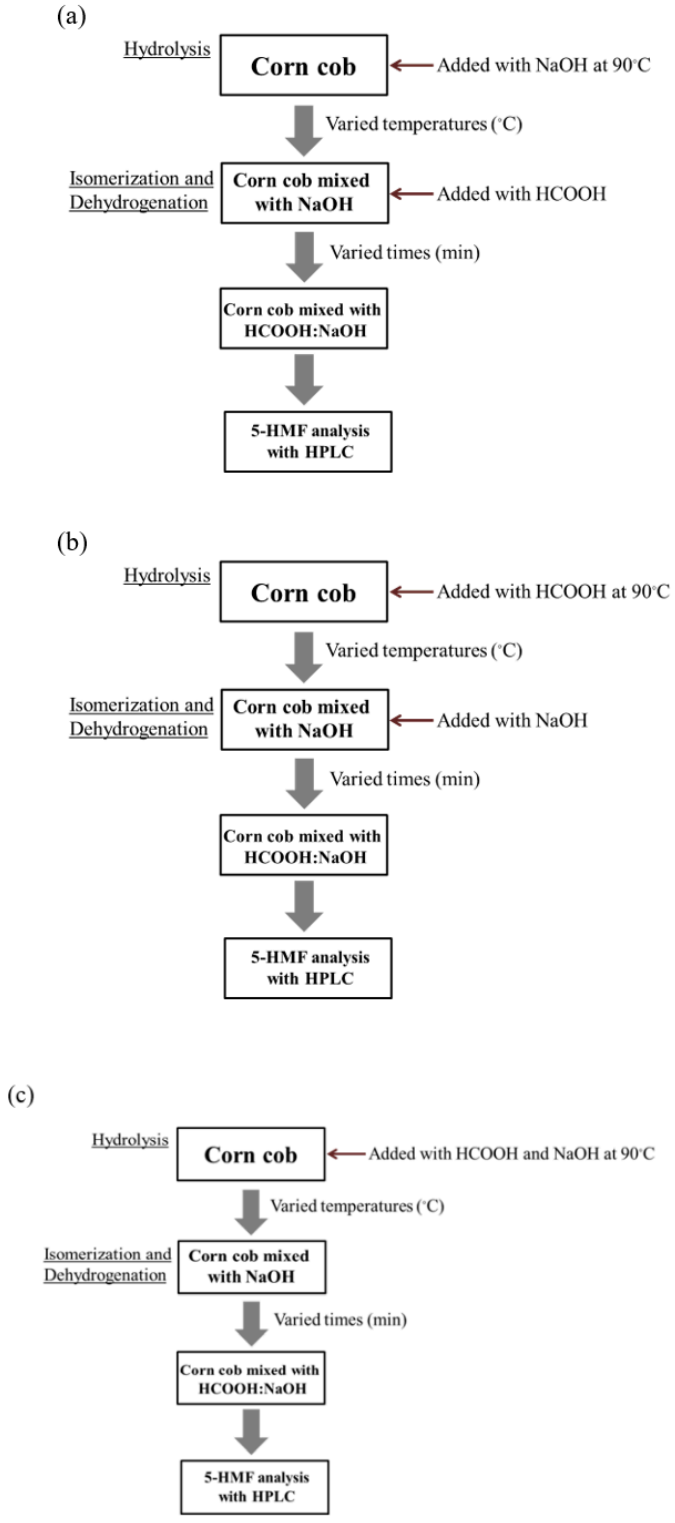


Figure 1

The preparation of green chemistry reaction contained with hydrolysis, isomerization and dehydrogenation of (a) method 1, (b) method 2 and (c) method 3

Note: The temperatures varied with 100°C, 110°C, 120°C, 130°C and 140°C and the times varied with 0, 60, 120, 180 and 240 min are used for method 1 to 3 experiments.

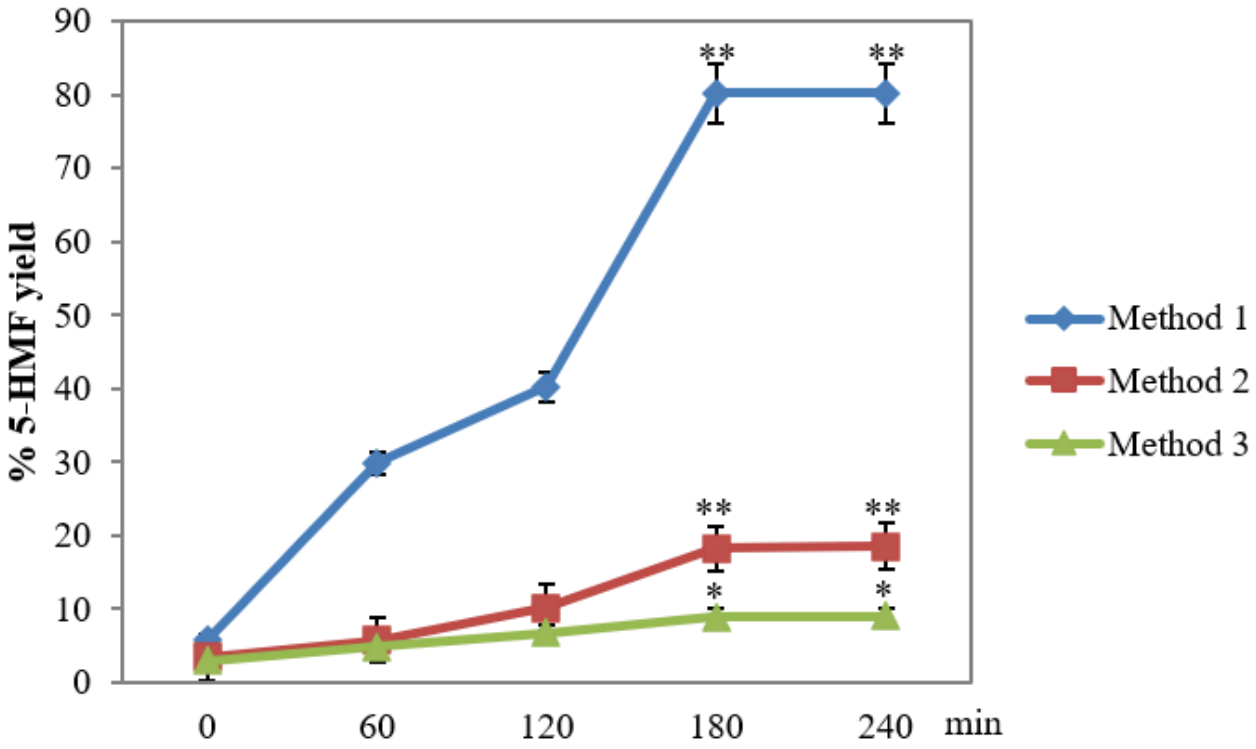


Figure 2

The percentage of 5-HMF yield of method 1, method 2 and method 3 at 120°C for 0, 60, 120, 180 and 240 min, n=3, $^{**}p \leq 0.01$ and $^{*}p \leq 0.05$.

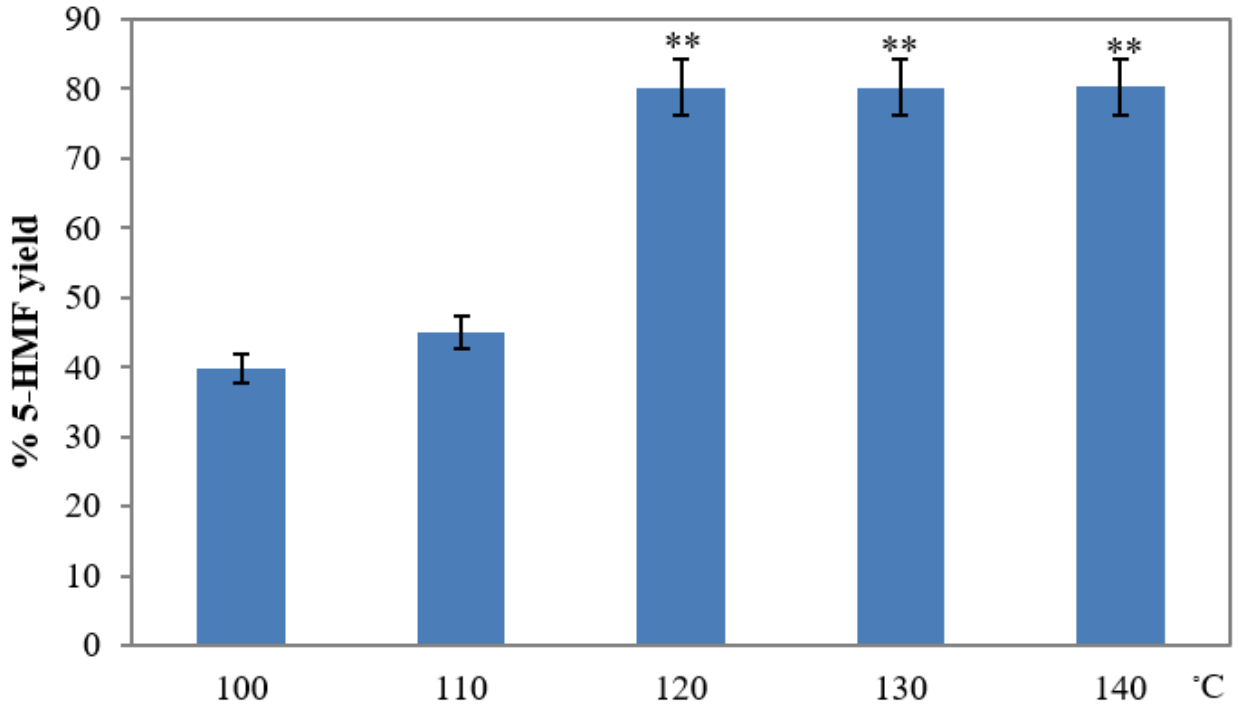


Figure 3

The percentage of 5-HMF of method 1 at 100°C, 110°C, 120°C, 130°C and 140 for 180 min, n= 3 and $^{**}p \leq 0.01$.

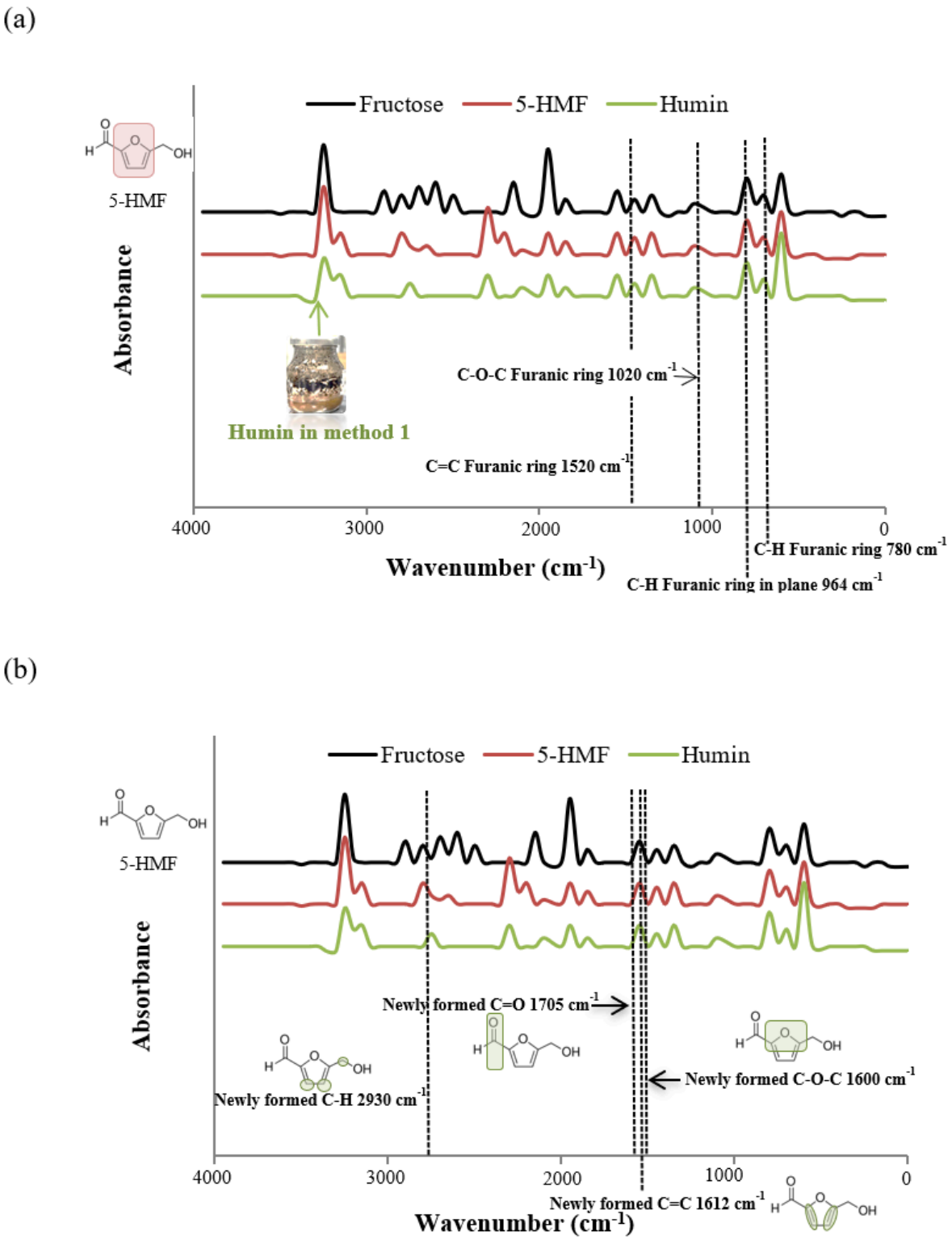


Figure 4

The overlay of FTIR spectra of fructose, 5-HMF and humin sample from the peaks of furan core and newly formed peaks are indicated with vertical lines.

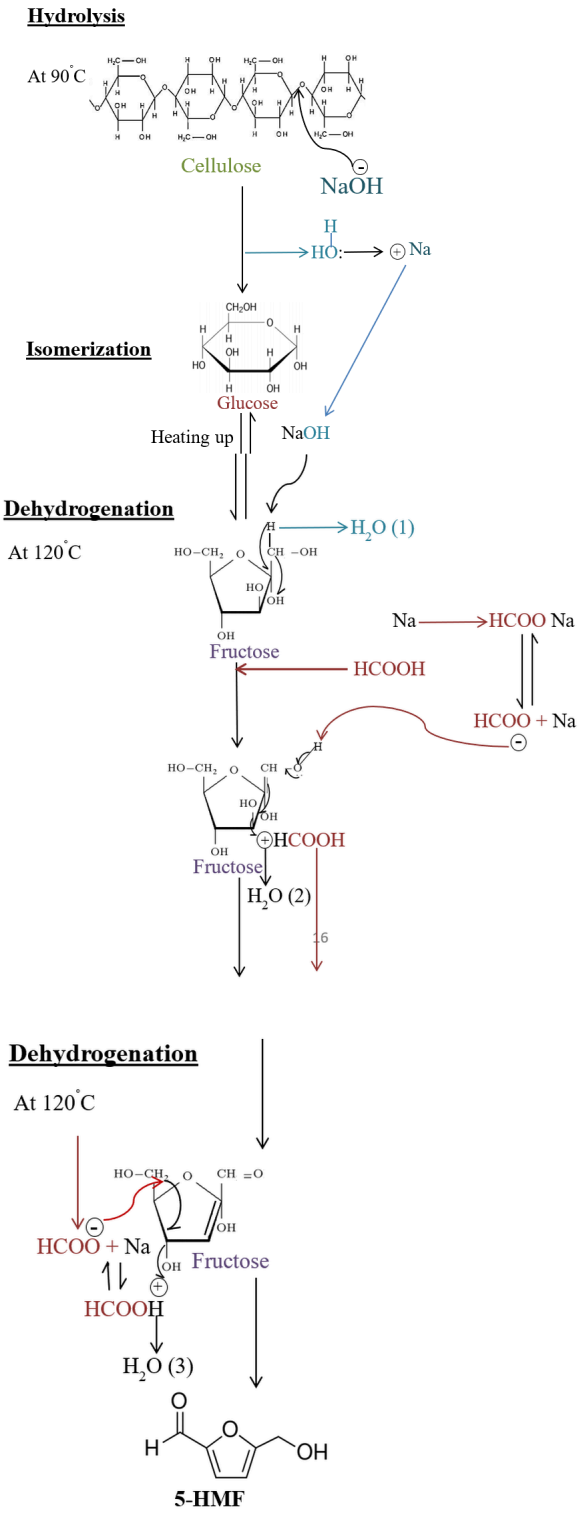


Figure 5

Mechanism of green chemistry reaction (corn cob mixed with HCOOH:NaOH) contained with 3 processes of (1) hydrolysis, (2) isomerization and (3) dehydrogenation at 120°C for 180 min of method 1

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- GraphicAbstract.docx