

A simple and efficient synthesis of 5-hydroxymethylfurfural from carbohydrates using acidic ionic liquid grafted on silica gel

Vinh Thanh Chau Doan,^{a, b, c} Thong Minh Dao,^{a, c} Thu Anh Huynh,^{a, c} The Thai Nguyen,^{a, c} and Phuong Hoang Tran^{*a, c}

^a Department of Organic Chemistry, Faculty of Chemistry, University of Science, Ho Chi Minh City, Vietnam.

^b Faculty of Interdisciplinary Science, University of Science, Ho Chi Minh City, Vietnam

^c Vietnam National University, Ho Chi Minh City, Vietnam

E-mail: thphuong@hcmus.edu.vn

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Section S1. Building a calibration curve of 5-HMF

To prepare the standard solution of 5-HMF, 10 mg of 5-HMF was dissolved in 10 mL of DI water to obtain the solution having a 1000 ppm concentration. Then, an exact amount of 1.0 mL of the 1000 ppm solution was taken out and diluted with DI water to 10 mL to prepare the 100 ppm solution. Next, various volumes of 100 ppm solution were taken out and diluted with DI water to form samples with concentrations of 1 to 10 ppm. These standard samples were analyzed by the UV-vis spectrometer on a JASCO V-670 UV-Vis double-beam spectrophotometer (Japan). The absorbance values were recorded at $\lambda_{\text{max}} = 285.0$ nm. The results are indicated in **Table S1**. Next, the calibration curve of 5-HMF was demonstrated as seen in **Fig. S1**.

Table S1: Standard concentrations of 5-HMF

Sample	Concentration (ppm)	Absorbance at 285.0 nm
1	0.94	0.157
2	1.89	0.234
3	3.78	0.472
4	5.66	0.673
5	7.55	0.841
6	9.44	1.005

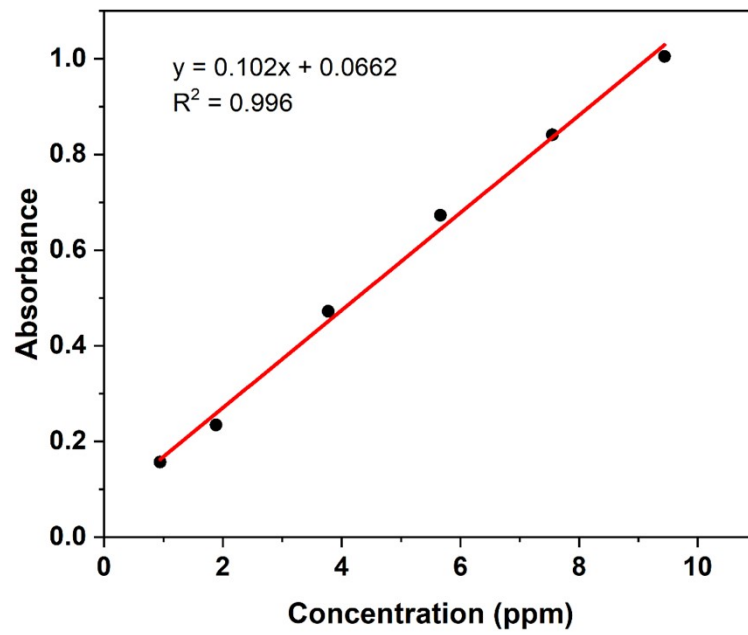


Figure S1: The calibration curve of 5-HMF at 285.0 nm

Section S2: HPLC and LC/MS/MS condition and the comparison between HPLC and UV-vis method

After completion of the reaction, approximately 0.1 g of the product mixture was taken out, diluted to 10 mL by DI water, and then filtered with a 0.45 μm membrane nylon filter before being analyzed with HPLC-DAD to calculate the yields of the products (5-HMF, 2,5-diformylfuran (DFF), and 2,5-furandicarboxylic acid (FDCA)). 5-HMF, DFF, and FDCA were detected by a C18 (4.6 \times 250 mm) column with trifluoroacetic acid/H₂O 0.05% flowing at 1.0 mL min⁻¹ as the mobile phase. The formulas to calculate 5-HMF, DFF, and FDCA yields were presented as follows:

$$\text{5-HMF yield (\%)} = [\text{moles of 5-HMF/moles of initial glucose}] \times 100$$

$$\text{DFF yield (\%)} = [\text{moles of DFF/moles of initial glucose}] \times 100$$

$$\text{FDCA yield (\%)} = [\text{moles of FDCA/moles of initial glucose}] \times 100$$

Glucose was detected by a PRP-X 100 Anion Exchange HPLC column (10 μm , 4.1 \times 250 mm) and an AB SCIEX Triple Quad 5500 LC/MS/MS System with electrospray ionization in negative ion mode with the temperature maintained at 25 $^{\circ}\text{C}$, and the mixture of acetonitrile and water flowing at 1.2 mL min⁻¹ as the mobile phase. The formulas to calculate glucose conversion and 5-HMF selectivity are:

$$\text{Glucose conversion (\%)} = 1 - [\text{mass of glucose in the mixture/mass of initial glucose}] \times 100$$

$$\text{5-HMF selectivity(\%)} = [\text{5-HMF yield/glucose conversion}] \times 100$$

$$\text{DFF selectivity(\%)} = [\text{DFF yield/glucose conversion}] \times 100$$

Additionally, we conducted an experiment involving fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), and DMSO (2 mL), heated at 120 $^{\circ}\text{C}$ for durations ranging from 0.5 to 4 h. The extracted samples were quantified using both UV-vis and HPLC methods and the results are denoted in Table S2. The relative errors for all samples ranged from 1.0 to 9.8%. Furthermore, no signals indicative of common alternative products with a similar ring structure to 5-HMF, such as DFF

or FDCA, were detected in the HPLC chromatographs. In conclusion, the UV-vis method can effectively determine the quantity of 5-HMF in the sample.

Table S2. 5-HMF yield by UV-vis and HPLC method and their relative error

Time (h)	5-HMF yield (UV-vis) (%)	5-HMF yield (HPLC) (%)	Relative error (%)
0.5	55.1	57.8	4.7
1	51.5	54.9	6.2
2	54.3	60.2	9.8
3	58.0	58.6	1.0
4	52.5	53.8	2.4

Section S3: FT-IR spectra of the original and silane chain grafted activated silica gel

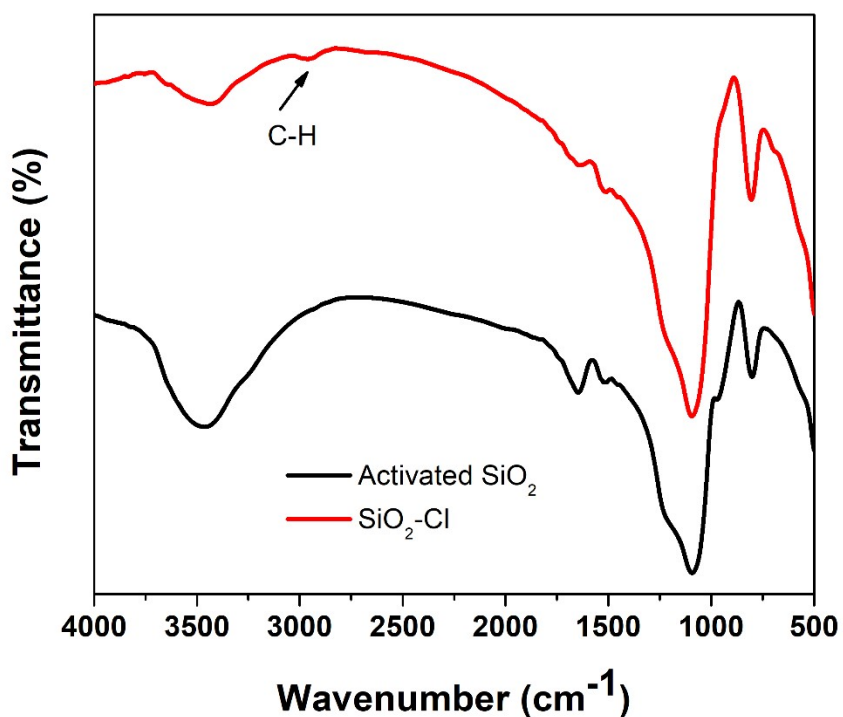


Figure S2: FT-IR spectra of the original and silane chain grafted activated silica gel

Section S4: The mass percentage of SiO₂-Imi-SO₃H-2 and recovered SiO₂-Imi-SO₃H-2 according to EDX

Table S3. The mass percentage of SiO₂-Imi-SO₃H-2 and recovered SiO₂-Imi-SO₃H-2 according to EDX

Element	SiO ₂ -Imi-SO ₃ H-2	Recovered SiO ₂ -Imi-SO ₃ H-2
C	15.53±0.10	15.20±0.10
N	0.43±0.03	0.15±0.04
O	39.91±0.16	49.77±0.20
Si	40.13±0.19	32.96±0.21
S	0.66±0.04	0.66±0.04
Cl	3.34±0.08	1.26±0.06
Total	100.00	100.00

Section S5: UV-vis spectra of the samples

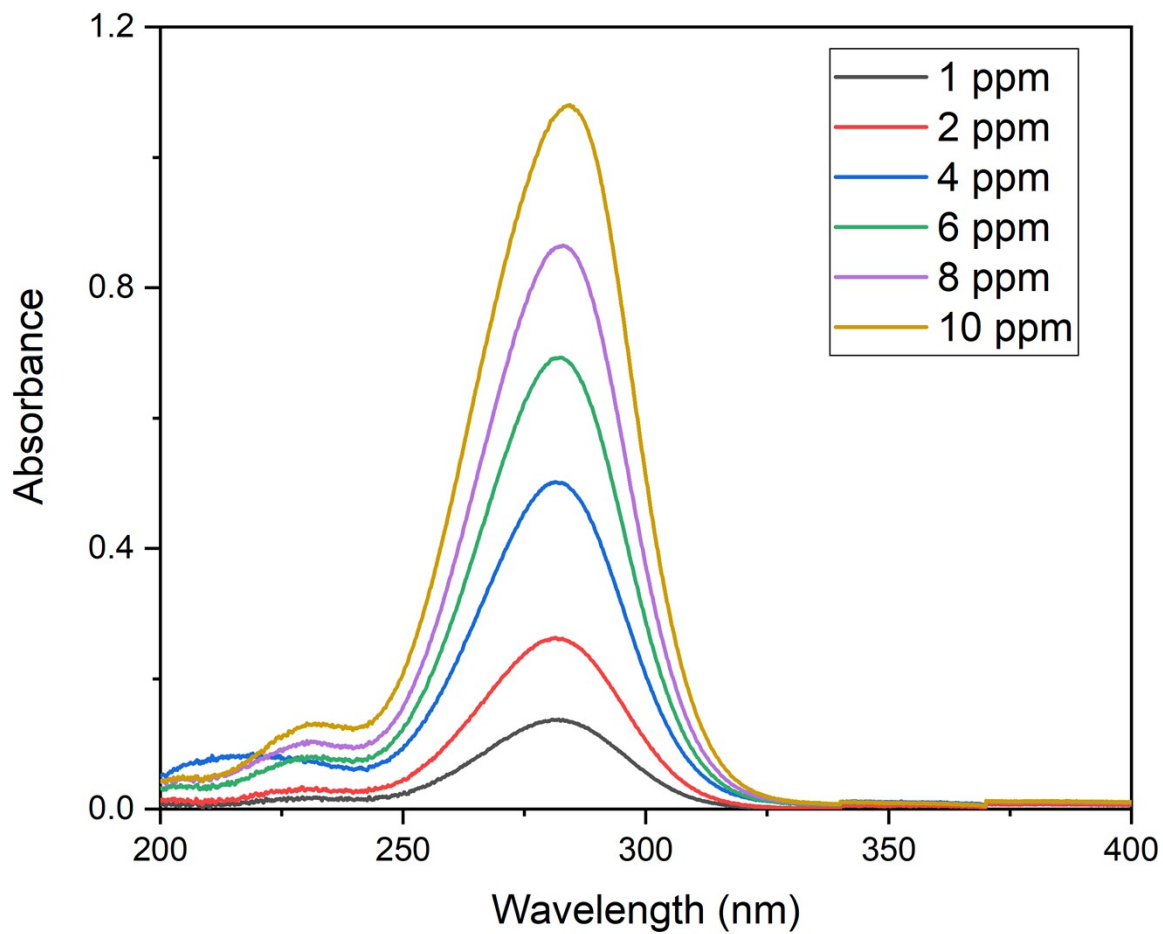


Figure S3.1. The UV-vis spectra of the calibration curve of 5-HMF

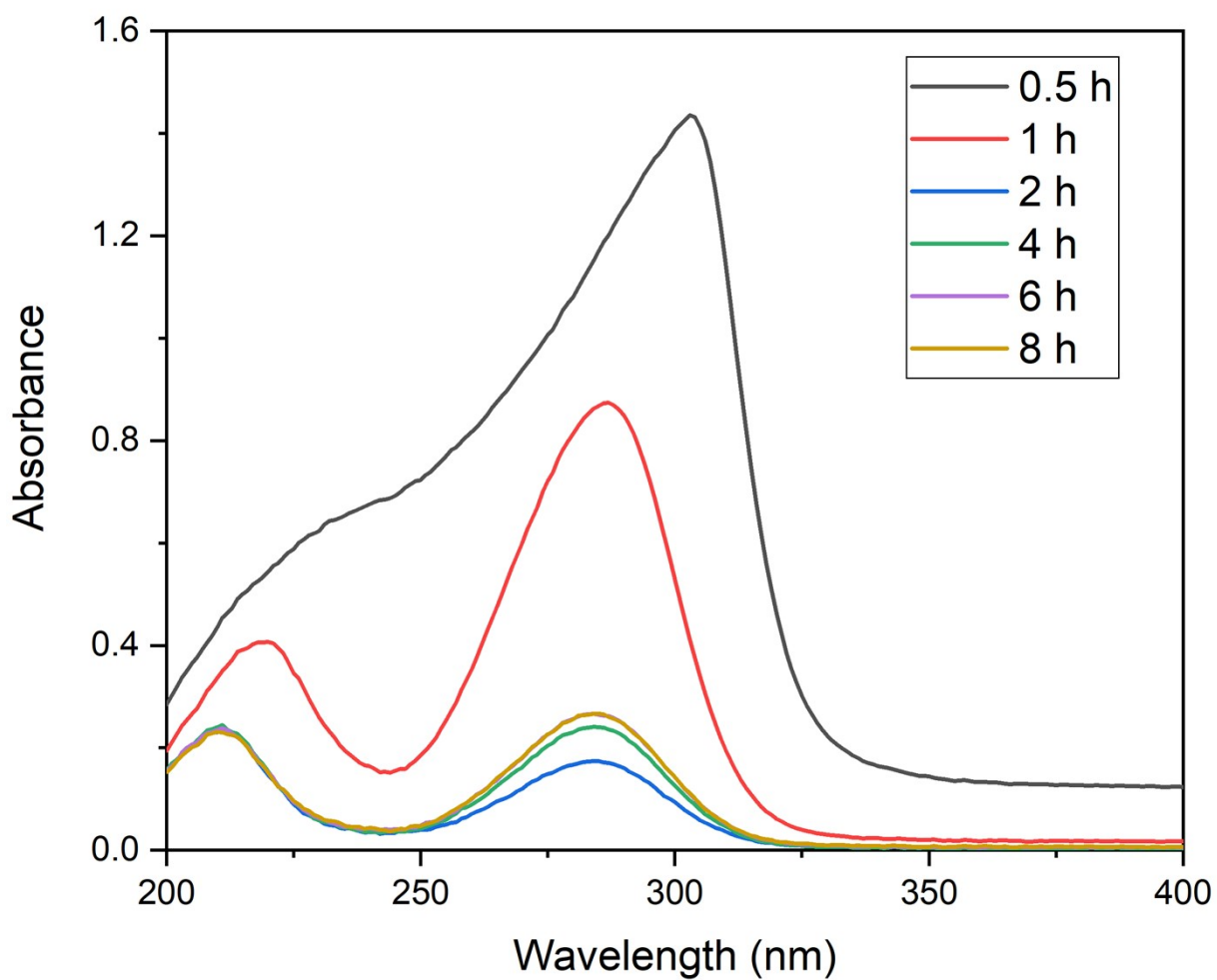


Figure S3.2. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), and SiO₂-Imi-SO₃H-2 of 5 mg, 120 °C, 8 h

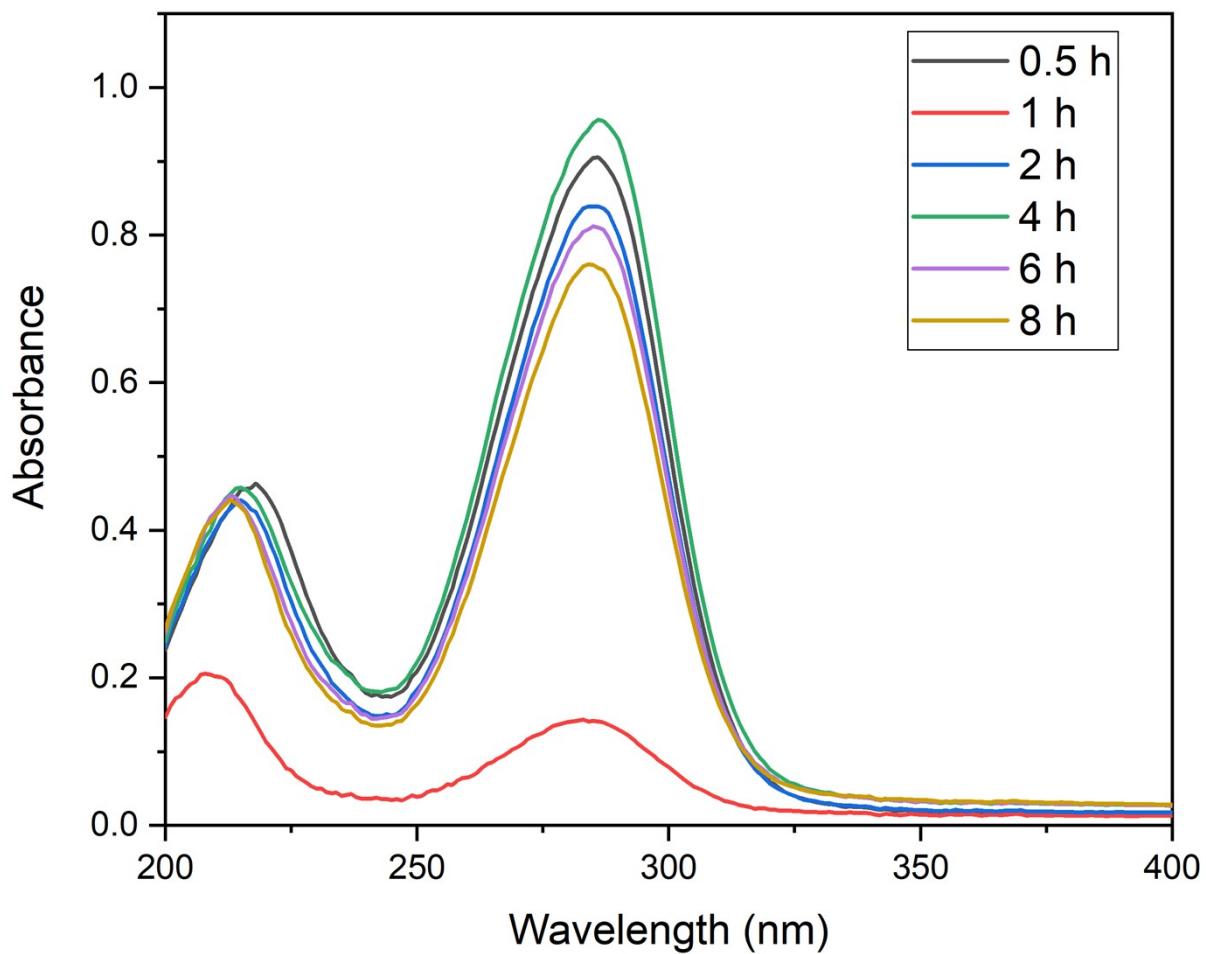


Figure S3.3. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), and SiO₂-Imi-SO₃H-2 of 10 mg, 120 °C, 8 h

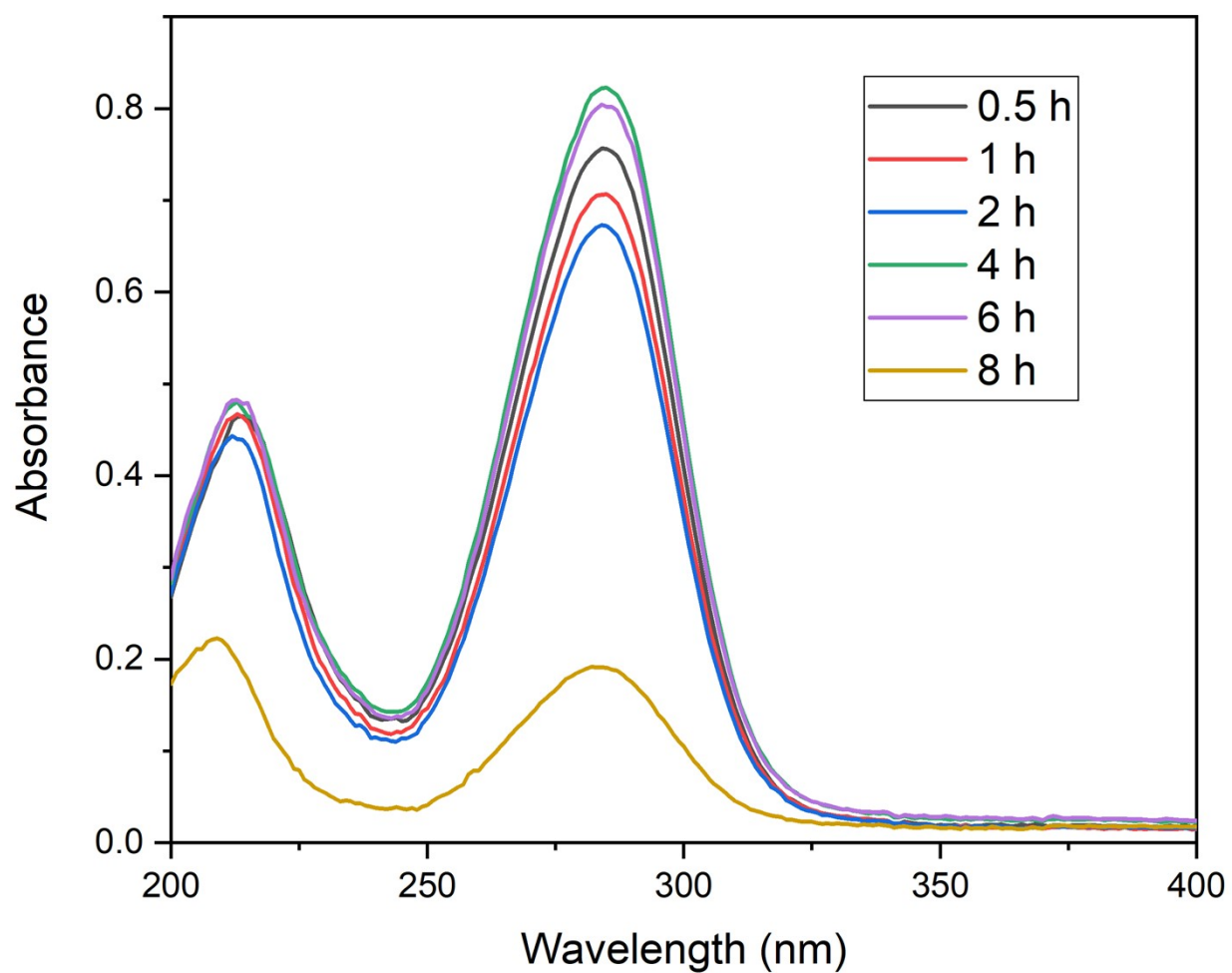


Figure S3.4. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), and SiO₂-Imi-SO₃H-2 of 30 mg, 120 °C, 8 h

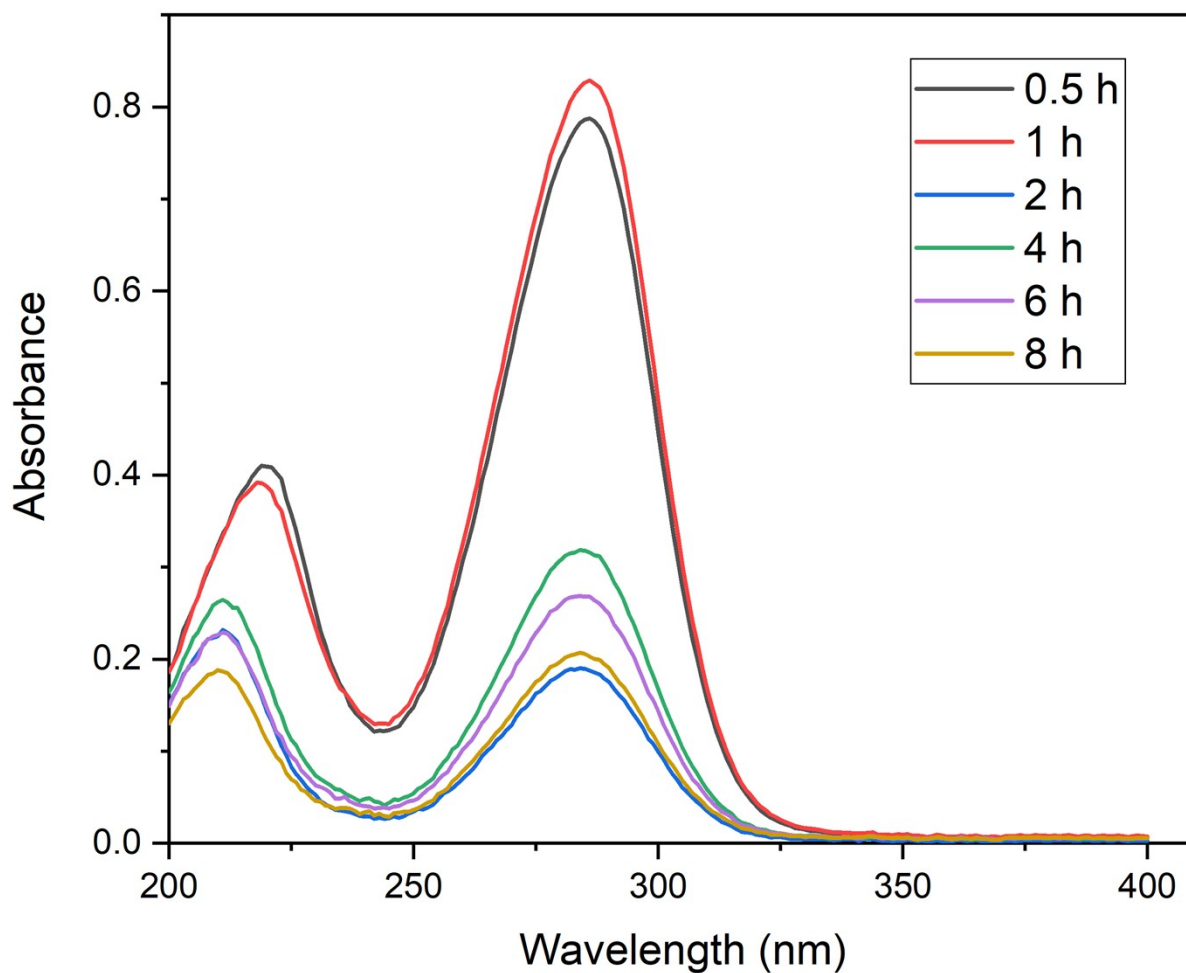


Figure S3.5. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), SiO₂-Imi-SO₃H-2 (20 mg), 120 °C, 8 h

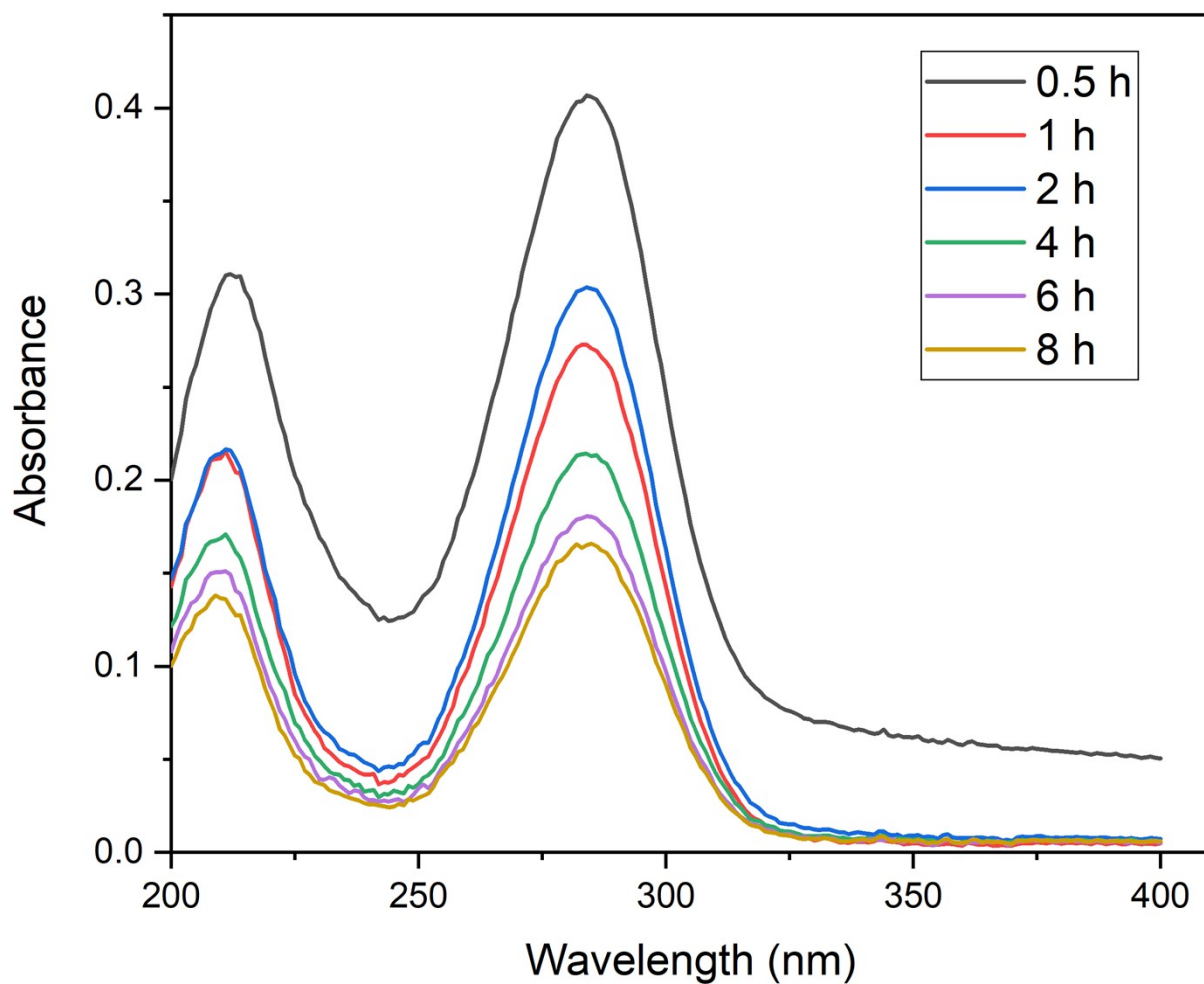


Figure S3.6. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), SiO₂-Imi-SO₃H-2 (20 mg), 140 °C, 8 h

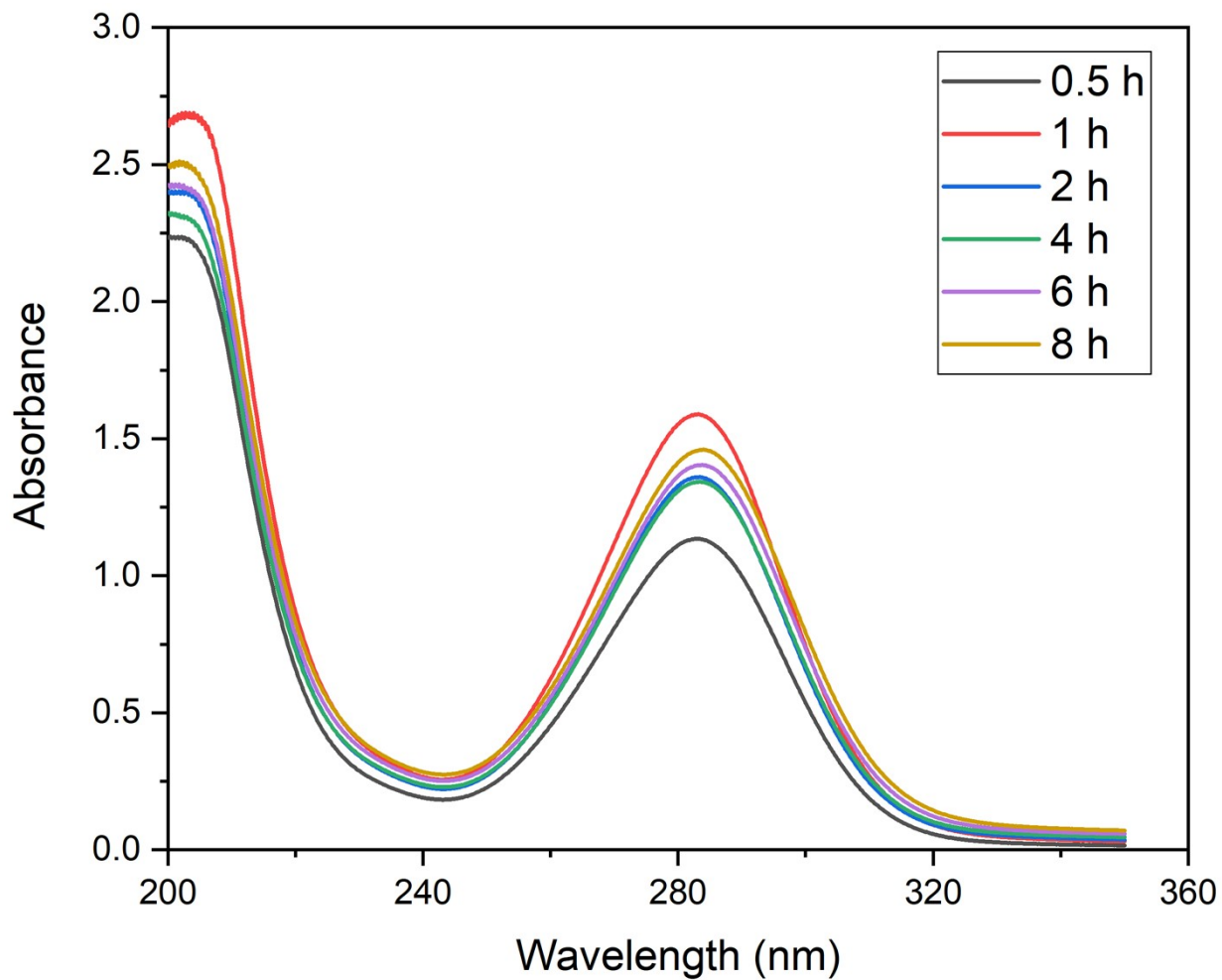


Figure S3.7. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), SiO₂-Imi-SO₃H-2 (20 mg), 160 °C, 8 h

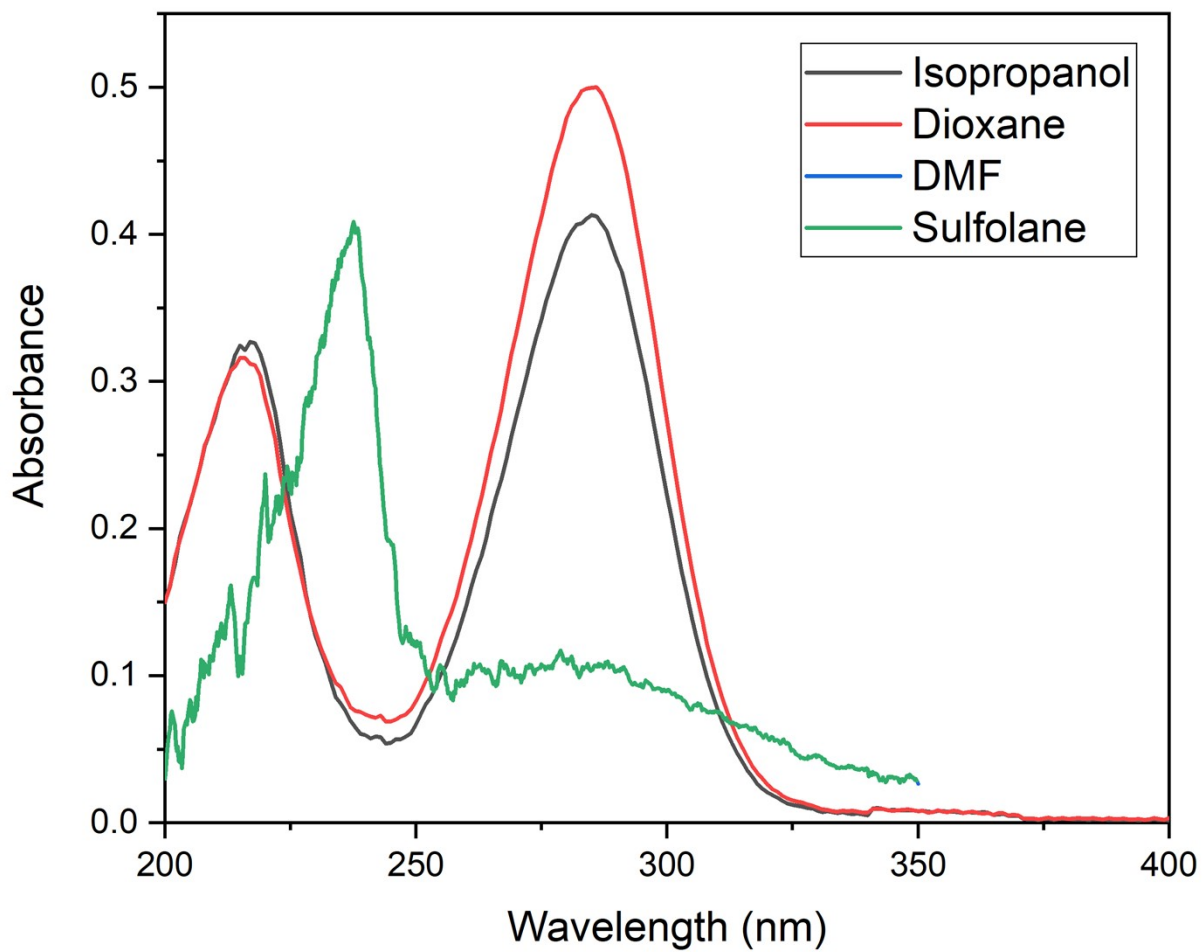


Figure S3.8. The UV-vis spectra of the samples of conditions: fructose (1 mmol), solvent (2 mL), SiO₂-Imi-SO₃H-2 (20 mg), 120 °C, 4 h

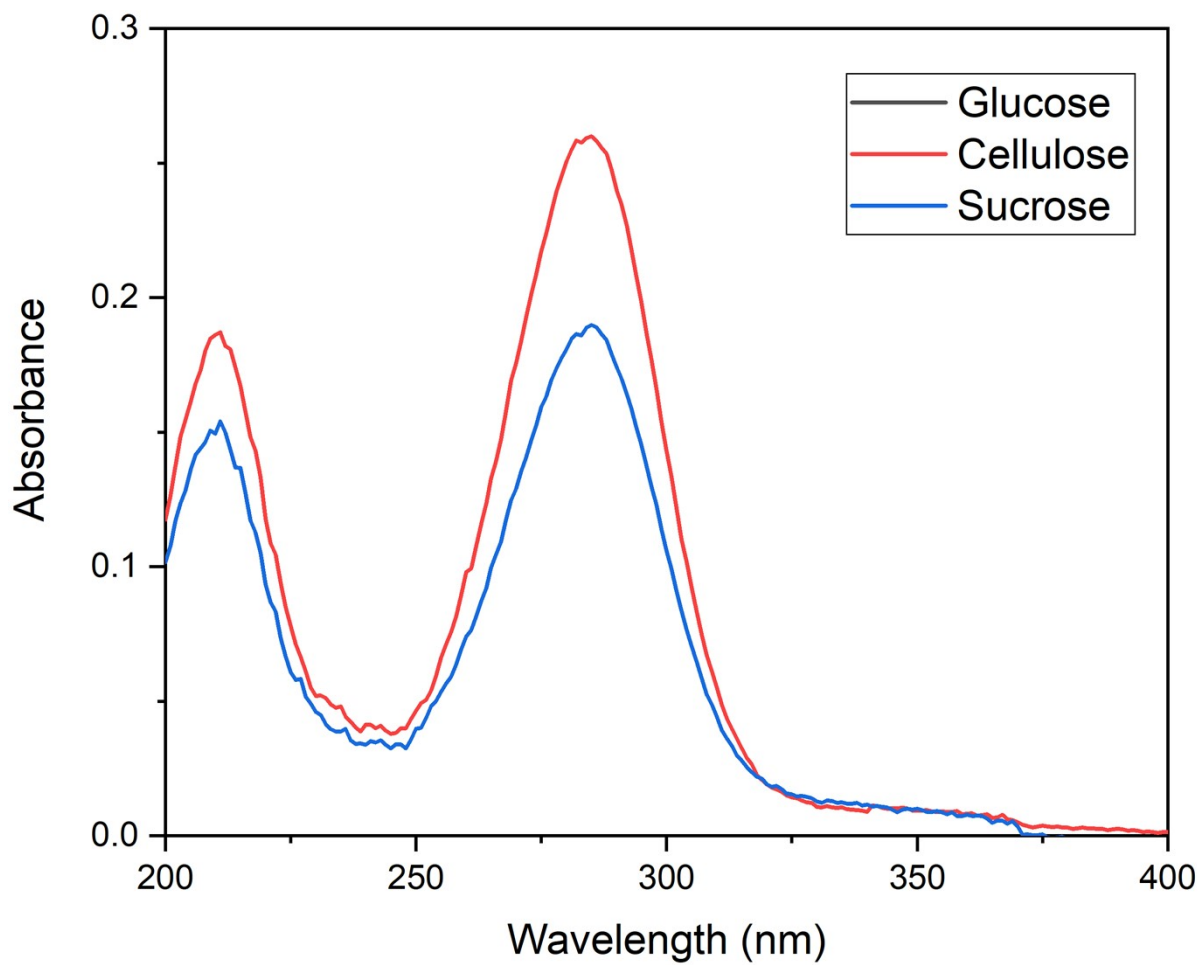


Figure S3.9. The UV-vis spectra of the samples of conditions: carbohydrate (1 mmol), solvent (2 mL), SiO₂-Imi-SO₃H-2 (20 mg), 120 °C, 4 h

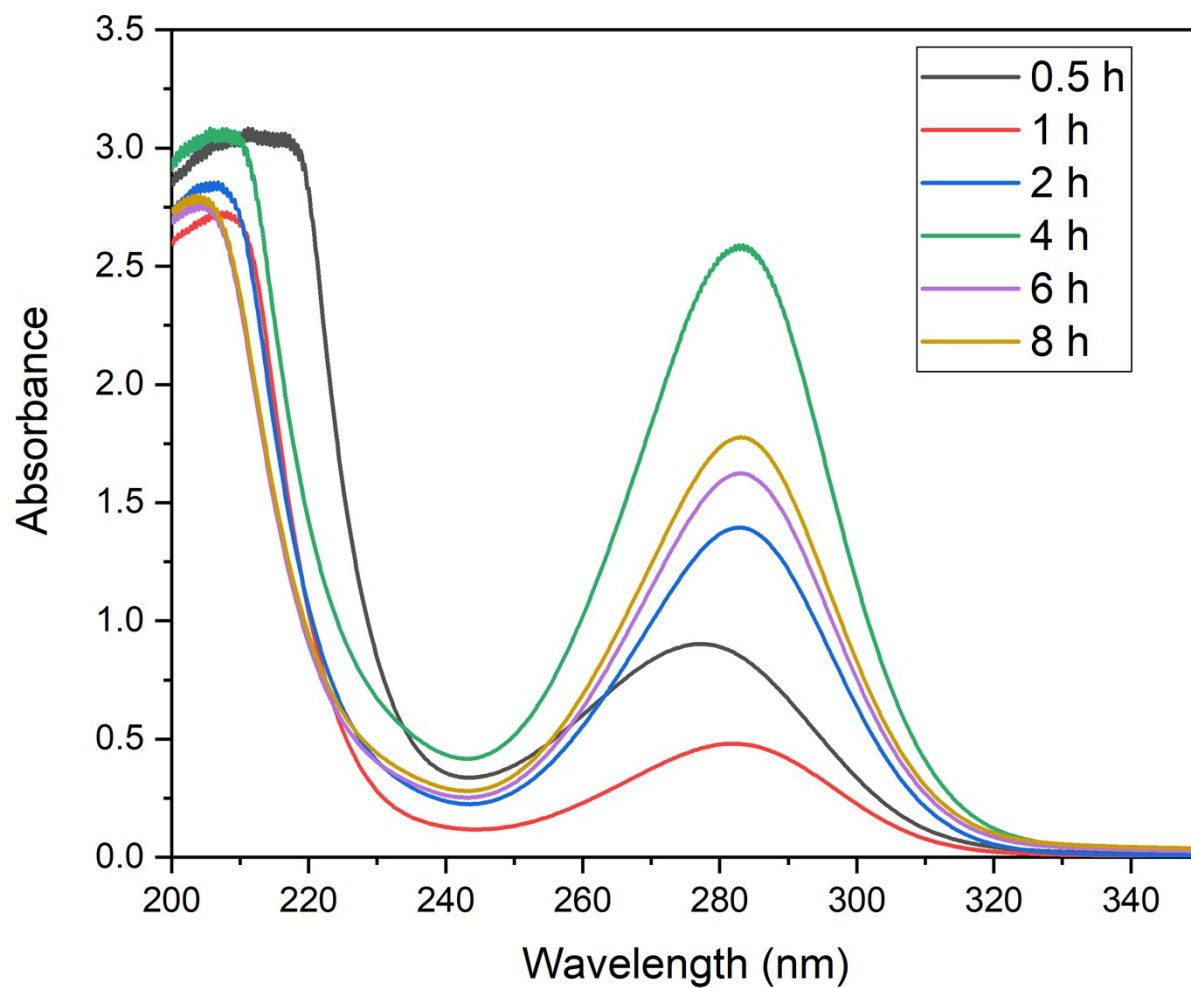


Figure S3.10. The UV-vis spectra of the samples of conditions: fructose (1 mmol), DMSO (2 mL), 120 °C, 8 h in the absence of a catalyst.

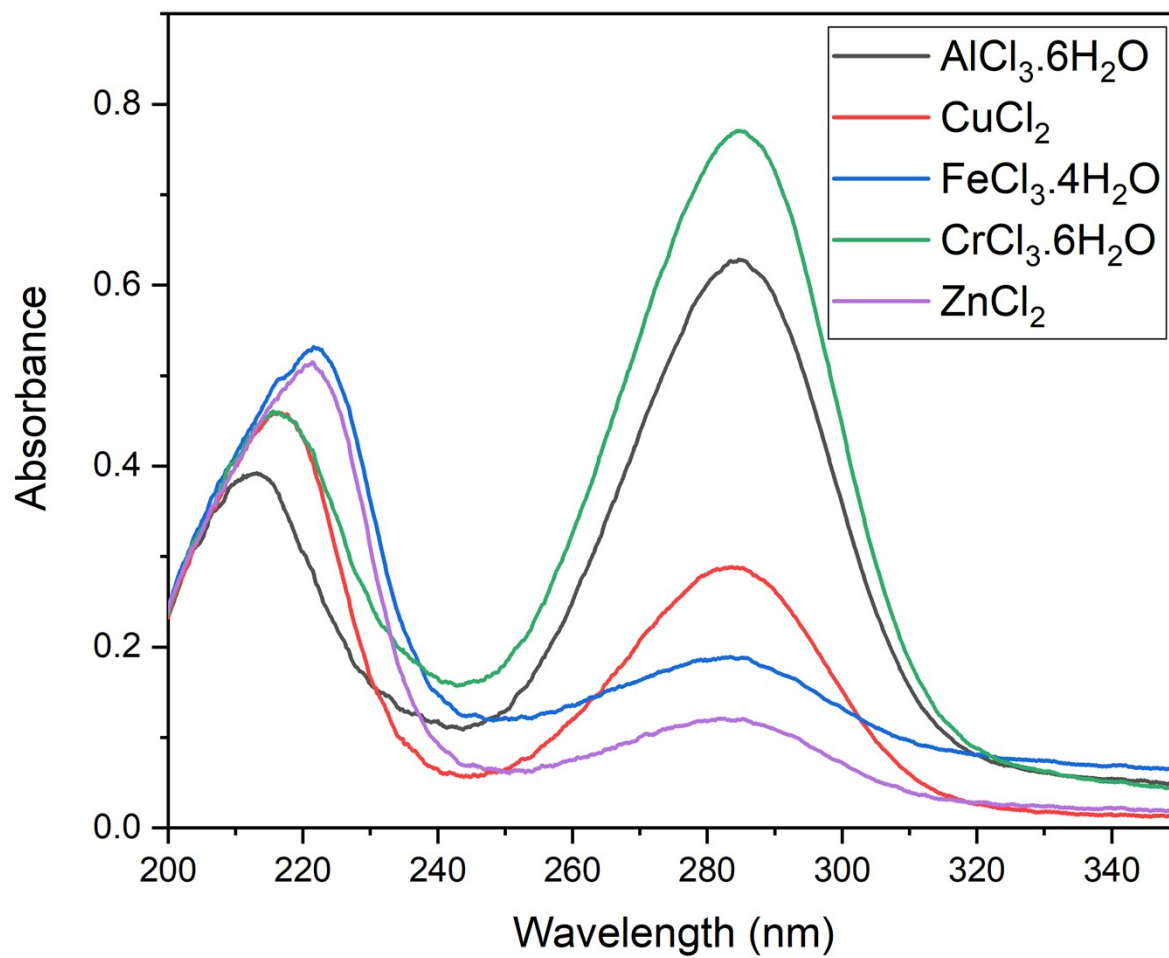


Figure S3.11. The UV-vis spectra of the samples of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), metal salt (20 mg), DMSO (2 mL), 120 °C, 4 h

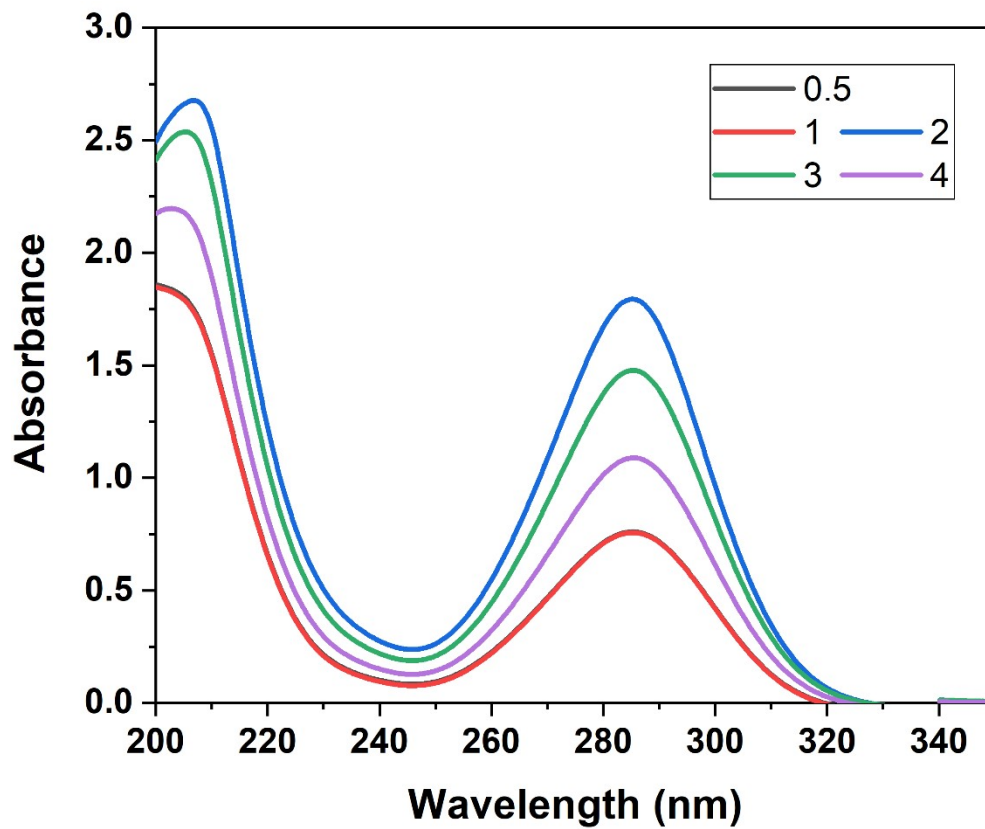


Figure S3.12. The UV-vis spectra of the samples of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 4 h

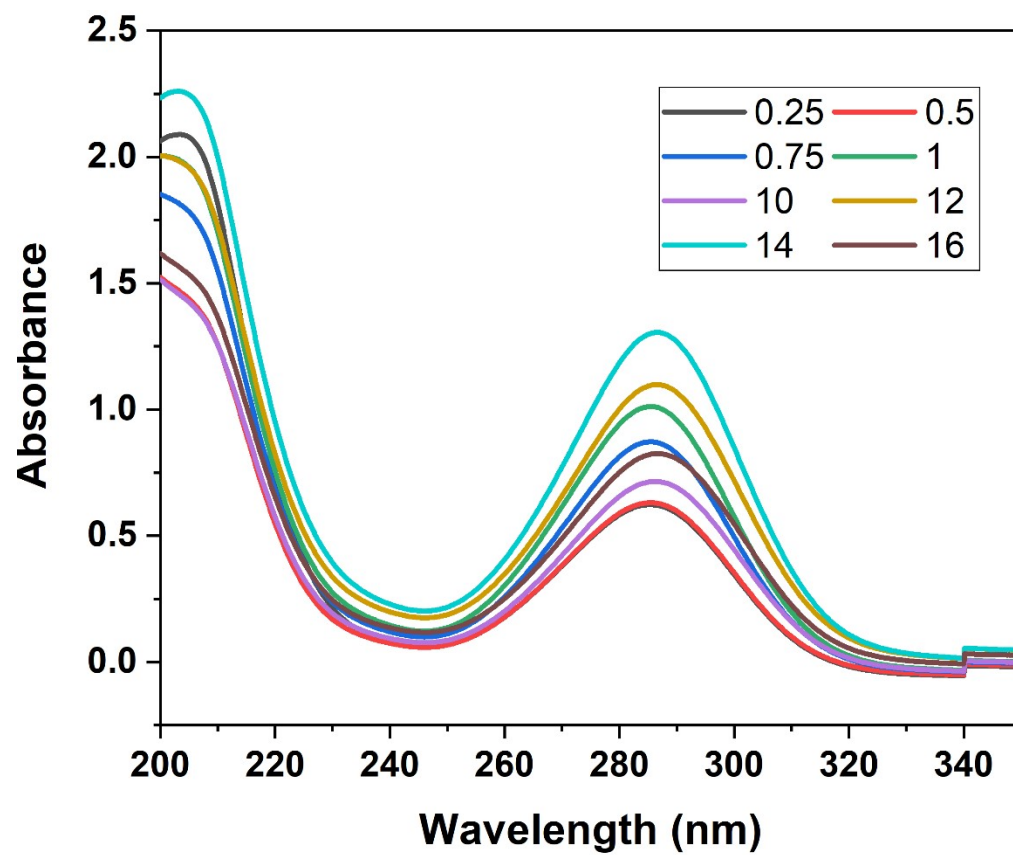


Figure S3.13. The UV-vis spectra of the samples of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 140 °C, 16 h

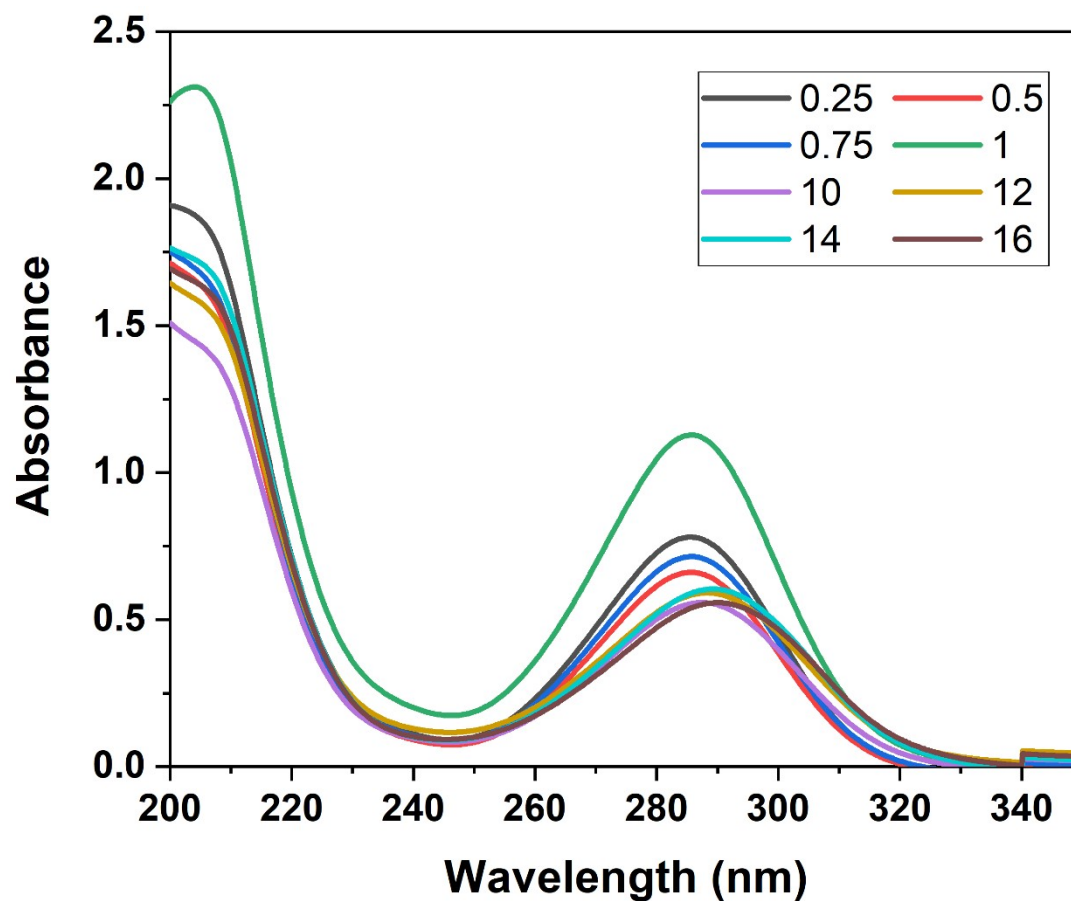


Figure S3.13. The UV-vis spectra of the samples of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 160 °C, 16 h

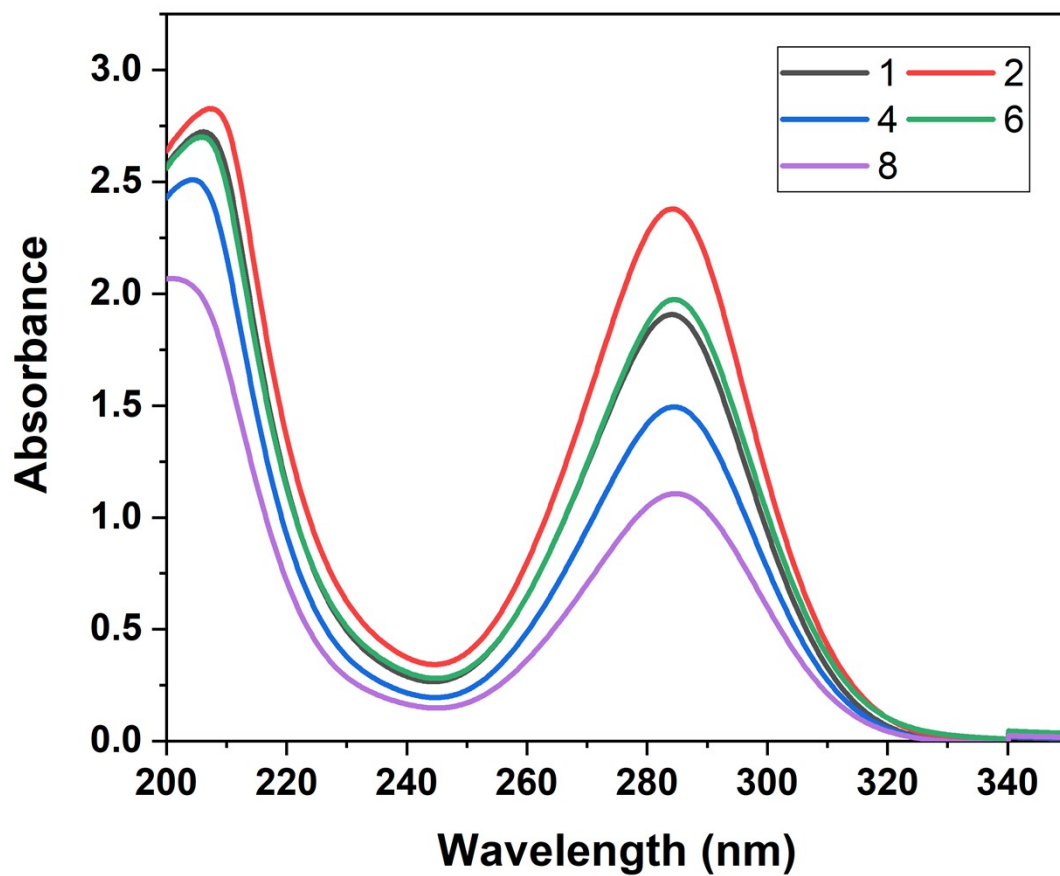


Figure S3.14. The UV-vis spectra of the samples of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-1 (20 mg), DMSO (2 mL), 120 °C, 8 h

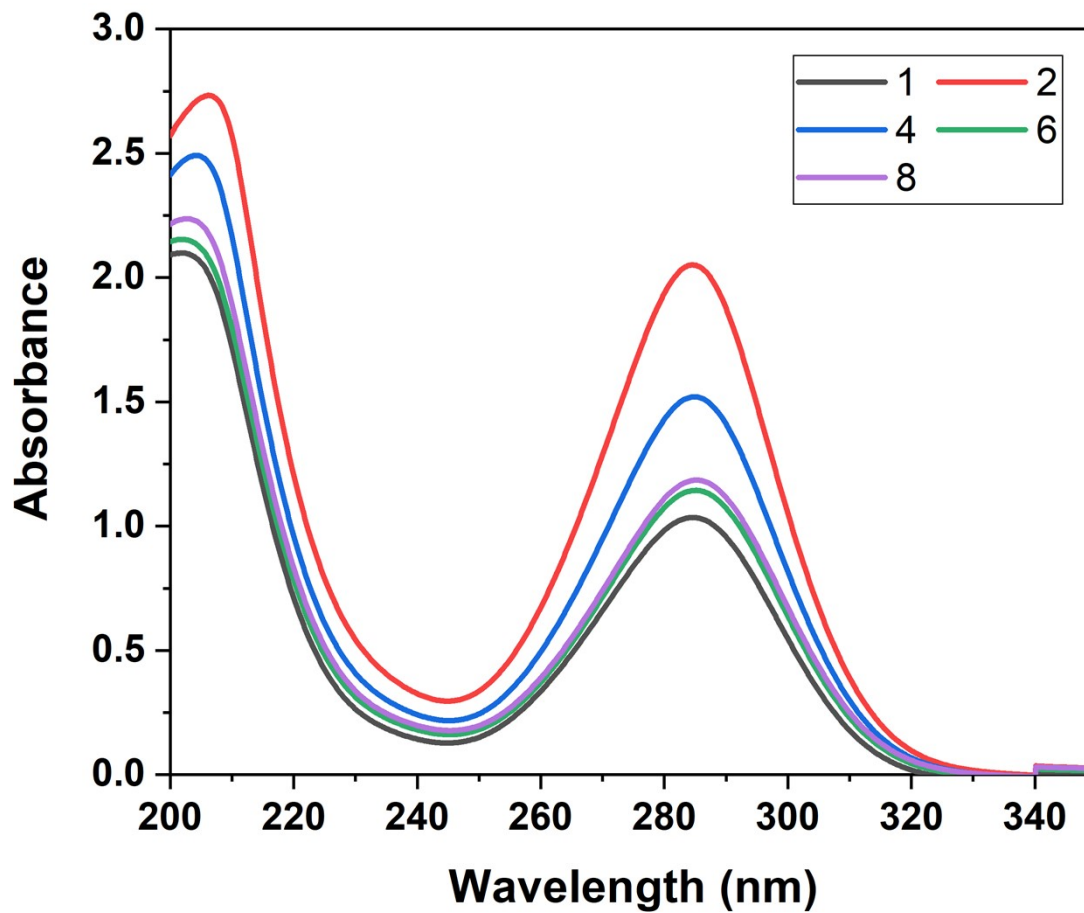
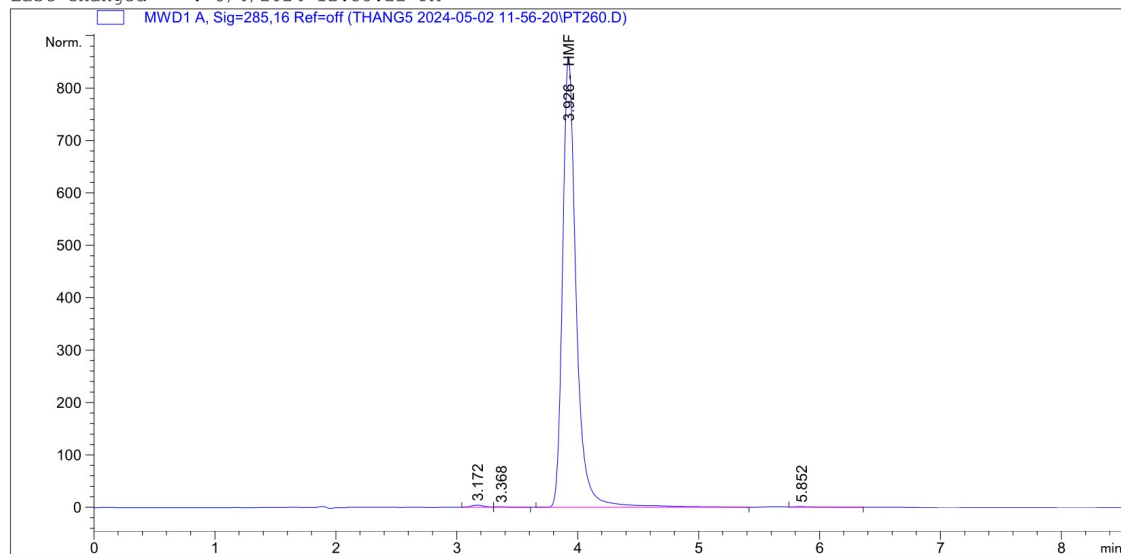


Figure S3.15. The UV-vis spectra of the samples of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-3 (20 mg), DMSO (2 mL), 120 °C, 8 h

Section S6: HPLC chromatographs of the samples

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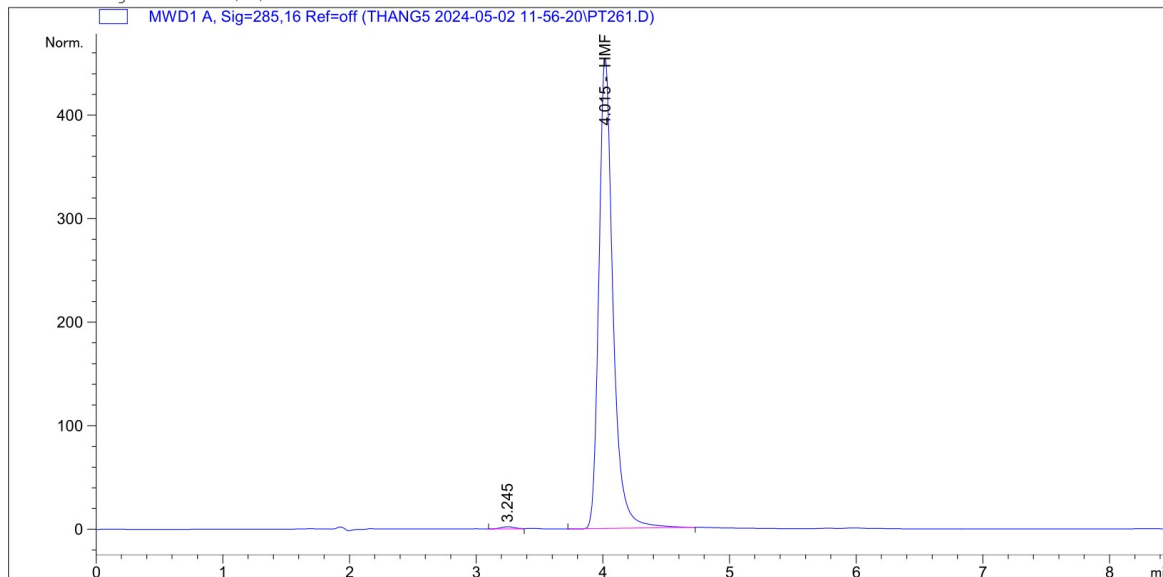
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2 Warnings or Errors :

Warning : Calibration warnings (see calibration table listing)
 Warning : Calibrated compound(s) not found

Figure S6.1. The HPLC chromatograph of the sample of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), AlCl₃.6H₂O (20 mg), DMSO (2 mL), 120 °C, 0.5 h

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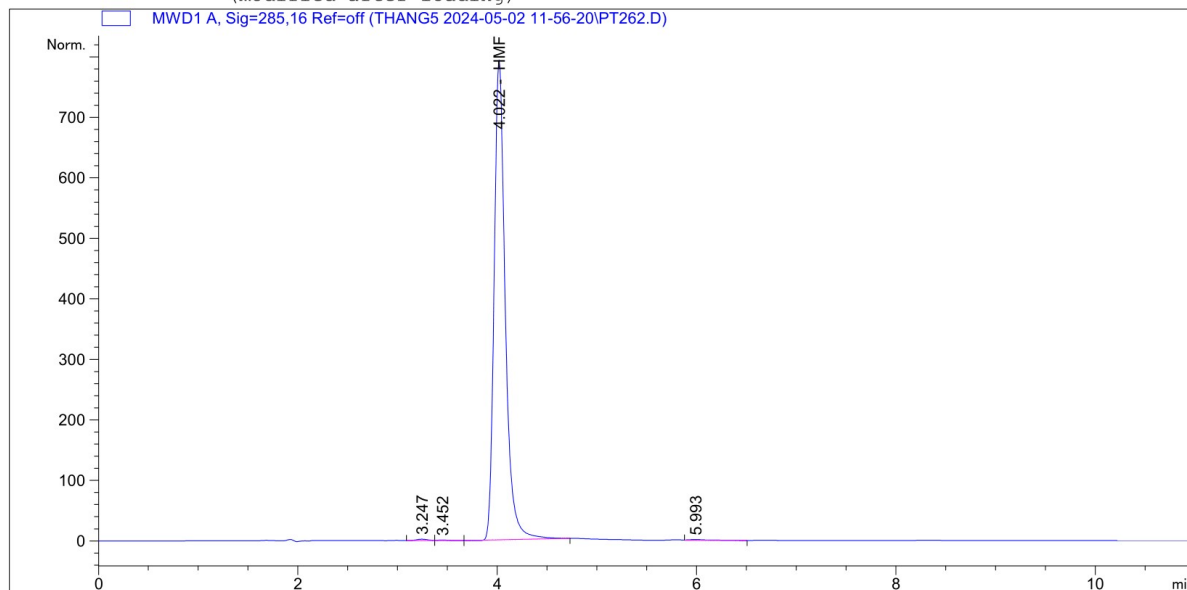
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Figure S6.2. The HPLC chromatograph of the sample of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), AlCl₃.6H₂O (20 mg), DMSO (2 mL), 120 °C, 1 h

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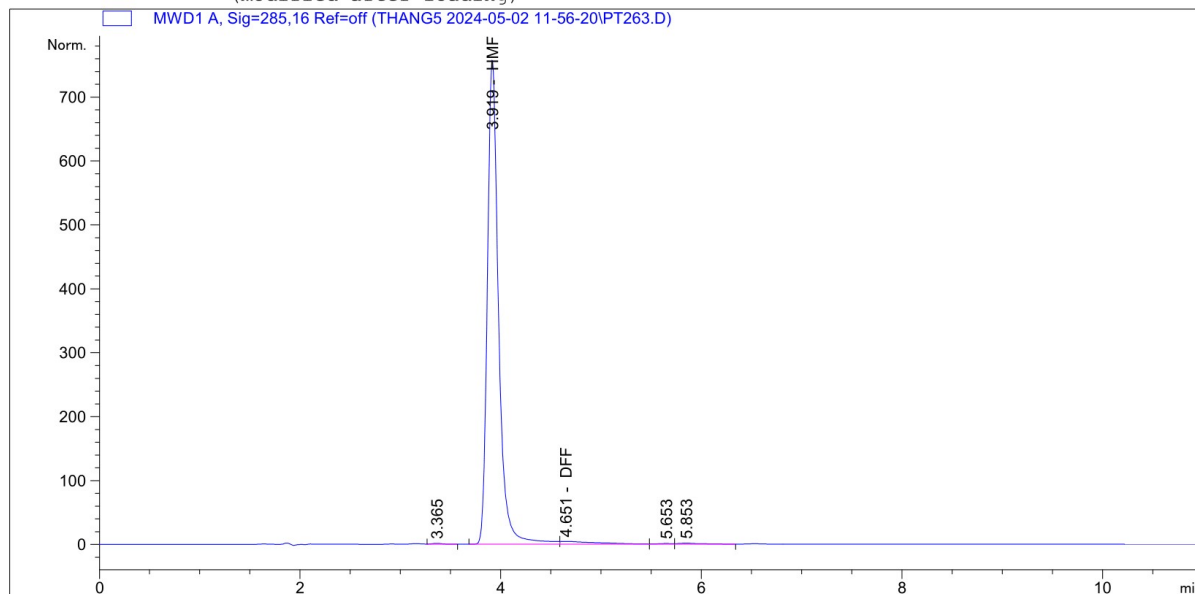
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Figure S6.3. The HPLC chromatograph of the sample of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), AlCl₃.6H₂O (20 mg), DMSO (2 mL), 120 °C, 2 h

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Totals : 177.14873

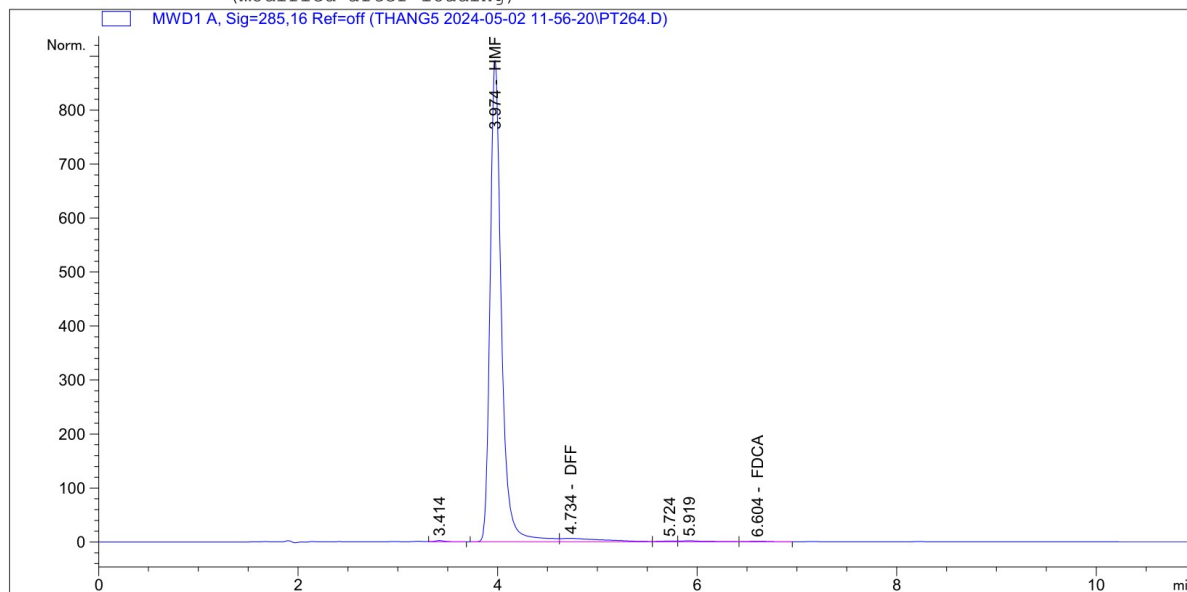
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Figure S6.4. The HPLC chromatograph of the sample of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), AlCl₃.6H₂O (20 mg), DMSO (2 mL), 120 °C, 3 h

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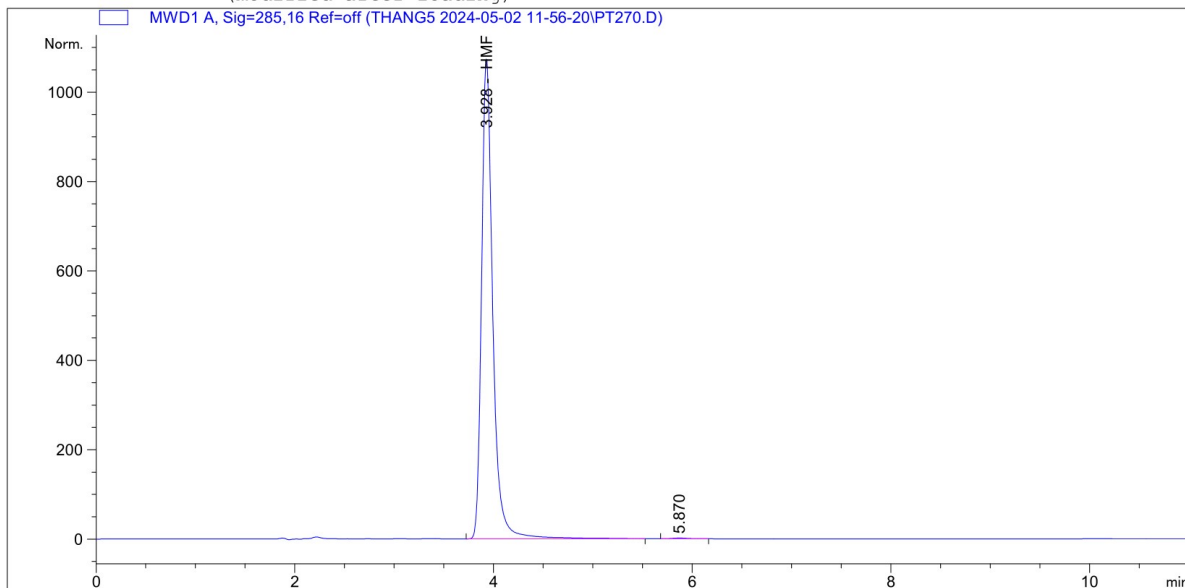
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Totals : 217.11973

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Figure S6.5. The HPLC chromatograph of the sample of conditions: glucose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), AlCl₃.6H₂O (20 mg), DMSO (2 mL), 120 °C, 4 h

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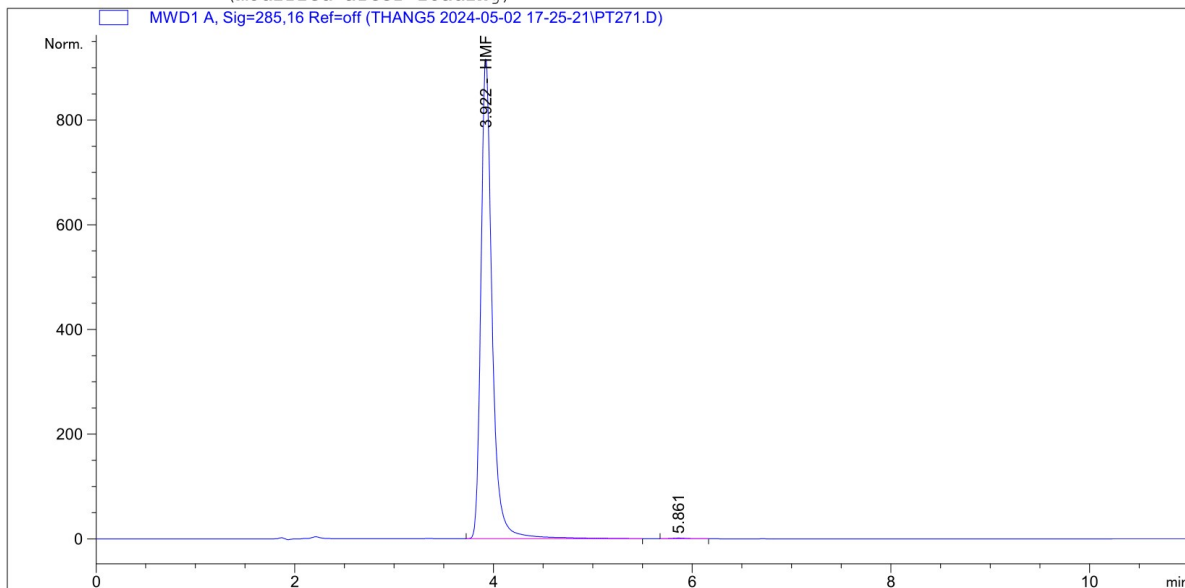
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Figure S6.6. The HPLC chromatograph of the sample of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 0.5 h

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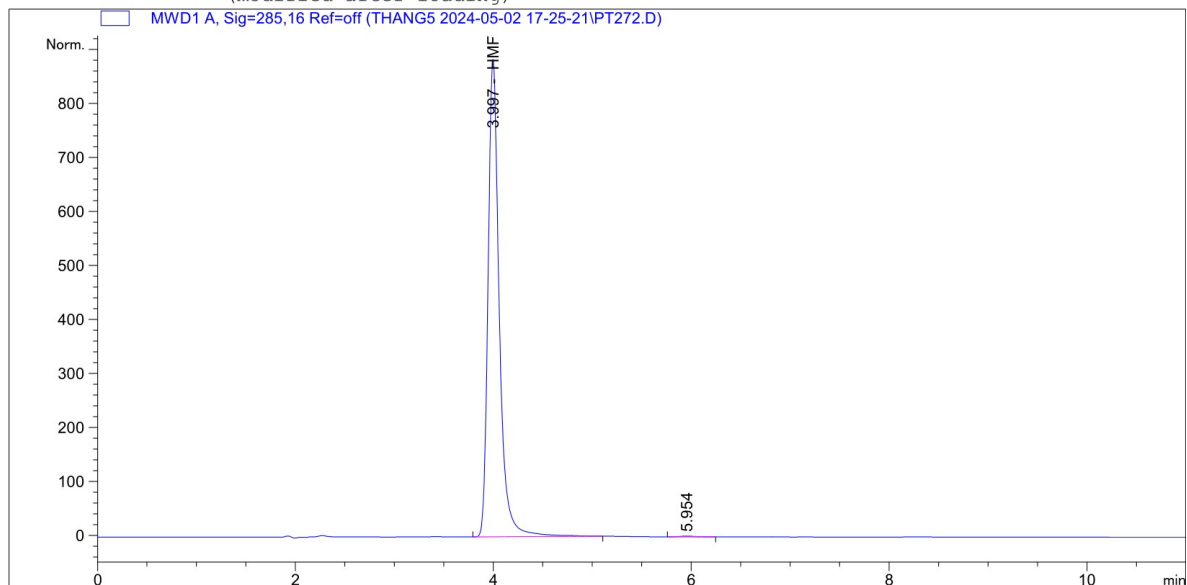
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Figure S6.7. The HPLC chromatograph of the sample of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 1 h

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Last changed    : 5/4/2024 12:36:58 PM
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External Standard Report

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Sorted By      :      Signal
Calib. Data Modified : 5/4/2024 12:34:49 PM
Multiplier:    :      1.0000
Dilution:     :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: MWD1 A, Sig=285,16 Ref=off

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ug/mL]	Grp	Name
3.997	BB	6947.52686	2.85083e-2	198.06186		HMF
4.711		-	-	-		DFE
6.689		-	-	-		FDCA

Totals : 198.06186

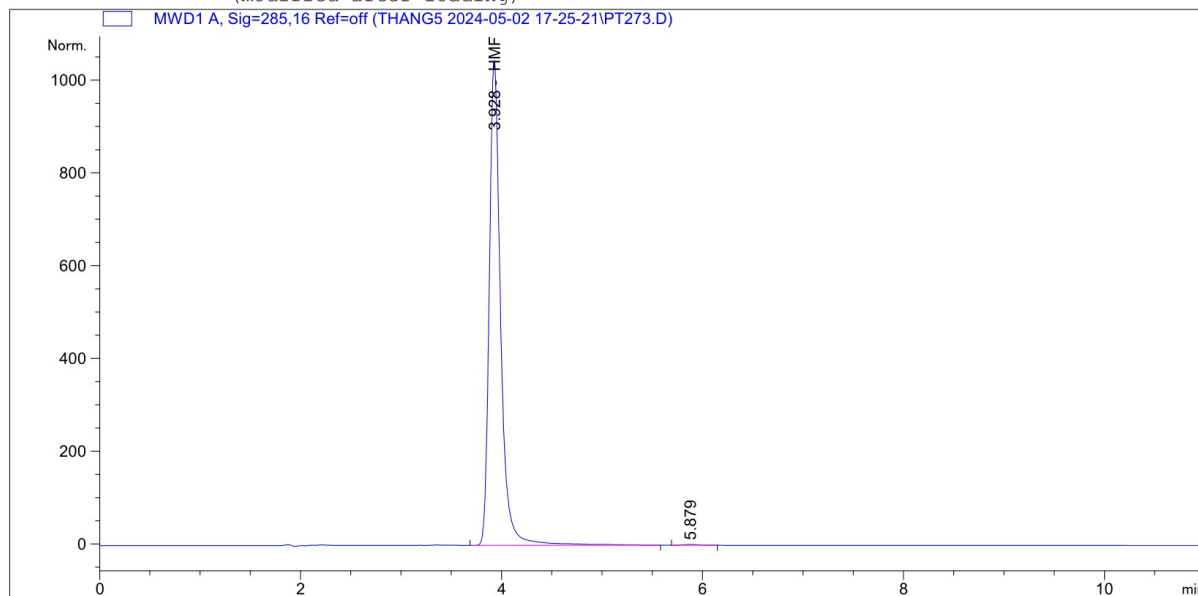
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

Figure S6.8. The HPLC chromatograph of the sample of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 2 h

Data File C:\CHEM32\1\DATA\THANG5 2024-05-02 17-25-21\PT273.D
Sample Name: PT273

```
=====
Acq. Operator   :                               Seq. Line :    3
Acq. Instrument : Instrument 1                   Location  : Vial 19
Injection Date  : 5/2/2024 5:54:21 PM           Inj       :    1
Acq. Method     : C:\CHEM32\1\DATA\HMF-DFF\THANG5 2024-05-02 17-25-21\THANH-VINH-TFA.M
Last changed    : 5/2/2024 5:24:13 PM
Analysis Method : C:\CHEM32\1\METHODS\VINH.M
Last changed    : 5/4/2024 12:36:58 PM
                (modified after loading)
=====
```



```
=====
External Standard Report
=====
```

```
Sorted By           :      Signal
Calib. Data Modified :      5/4/2024 12:34:49 PM
Multiplier          :            1.0000
Dilution            :            1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: MWD1 A, Sig=285,16 Ref=off

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ug/mL]	Grp	Name
3.928	VB	8263.45898	2.89725e-2	239.41291		HMF
4.711		-	-	-		DFF
6.689		-	-	-		FDCA

Totals : 239.41291

1 Warnings or Errors :

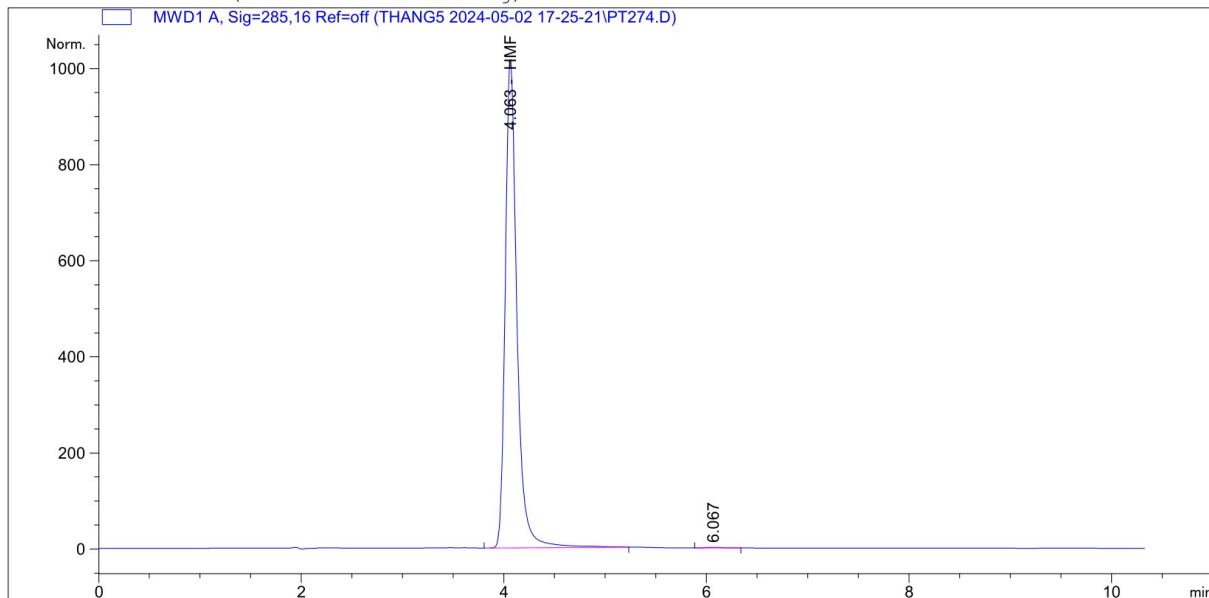
Warning : Calibrated compound(s) not found

Instrument 1 5/4/2024 1:09:33 PM

Page 1 of 2

Figure S6.9. The HPLC chromatograph of the sample of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 3 h

```
=====
Acq. Operator   :                               Seq. Line :    4
Acq. Instrument : Instrument 1                   Location  : Vial 20
Injection Date  : 5/2/2024 6:08:45 PM           Inj       :    1
Acq. Method     : C:\CHEM32\1\DATA\HMF-DFE\THANG5 2024-05-02 17-25-21\THANH-VINH-TFA.M
Last changed    : 5/2/2024 5:24:13 PM
Analysis Method : C:\CHEM32\1\METHODS\VINH.M
Last changed    : 5/4/2024 12:36:58 PM
                  (modified after loading)
=====
```



External Standard Report

```
Sorted By      :      Signal
Calib. Data Modified : 5/4/2024 12:34:49 PM
Multiplier:    :      1.0000
Dilution:      :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: MWD1 A, Sig=285,16 Ref=off

RetTime [min]	Type	Area [mAU*s]	Amt/Area	Amount [ug/mL]	Grp	Name
4.063	VB	8144.87061	2.89368e-2	235.68646		HMF
4.711		-	-	-		DFE
6.689		-	-	-		FDCA

Totals : 235.68646

1 Warnings or Errors :

Warning : Calibrated compound(s) not found

Figure S6.10. The HPLC chromatograph of the sample of conditions: fructose (1 mmol), SiO₂-Imi-SO₃H-2 (20 mg), DMSO (2 mL), 120 °C, 4 h

