

Electronic supplementary information (ESI)

A green approach for preparation of surfactant embedded sulfonated carbon catalyst towards glycerol acetalization reaction

Anindya Ghosh,^a Aniruddha Singha,^a Aline Auroux,^b Avik Das,^{c,d} Debasis Sen,^{c,d} and Biswajit Chowdhury^{*,a}

^a Department of Chemistry, Indian Institute of Technology (Indian School of Mines), Dhanbad, Jharkhand-826004, India.

^b Institut de recherches sur la catalyse et l'environnement de Lyon (IRCELYON), UMR 5256 CNRS- Université Lyon1, 2 avenue Albert Einstein, 69626, Villeurbanne Cedex, France.

^c Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai-400085, India.

^d Homi Bhabha National Institute, Mumbai-400094, India.

* Corresponding author. Tel.: (+91)-326-2235663; Fax.: (+91)-326-2296563; E-mail:
biswajit72@iitism.ac.in.

<u>Section</u>	<u>Contents</u>	<u>Page numbers</u>
Section S1	FESEM images and corresponding particle size distributions	S2
Section S2	XPS and CHNS/O analysis	S3-S5
Section S3	Effect of different reaction parameters	S6-S7
Section S4	Macromolecules formation mechanism	S8
Section S5	XRD, N ₂ adsorption/desorption, FTIR, ¹³ C-NMR, XPS, and Raman analysis of GSR-SO ₃ Hcatalyst	S9-S12
Section S6:	Calculation of atomic percentage to mmol g ⁻¹	S12

Section S1: FESEM images and corresponding particle size distributions

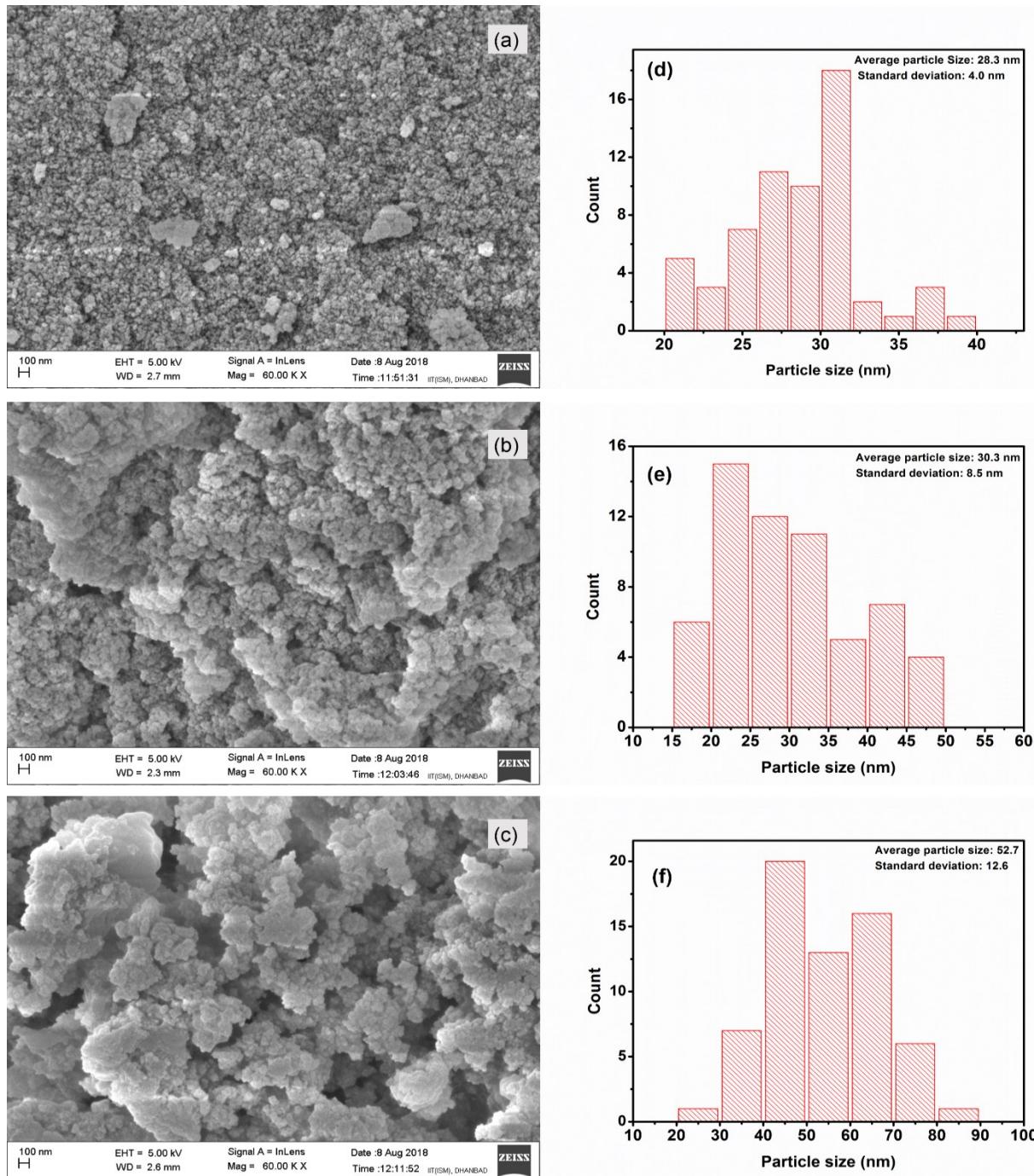


Figure S1. FESEM images (100 nm scale) of (a) G-SO₃H, (b) GL-SO₃H, and (c) GS-SO₃H catalysts. Images (d) to (f) show their respective particle size distributions (histograms).

Section S2: XPS and CHNS/O analysis

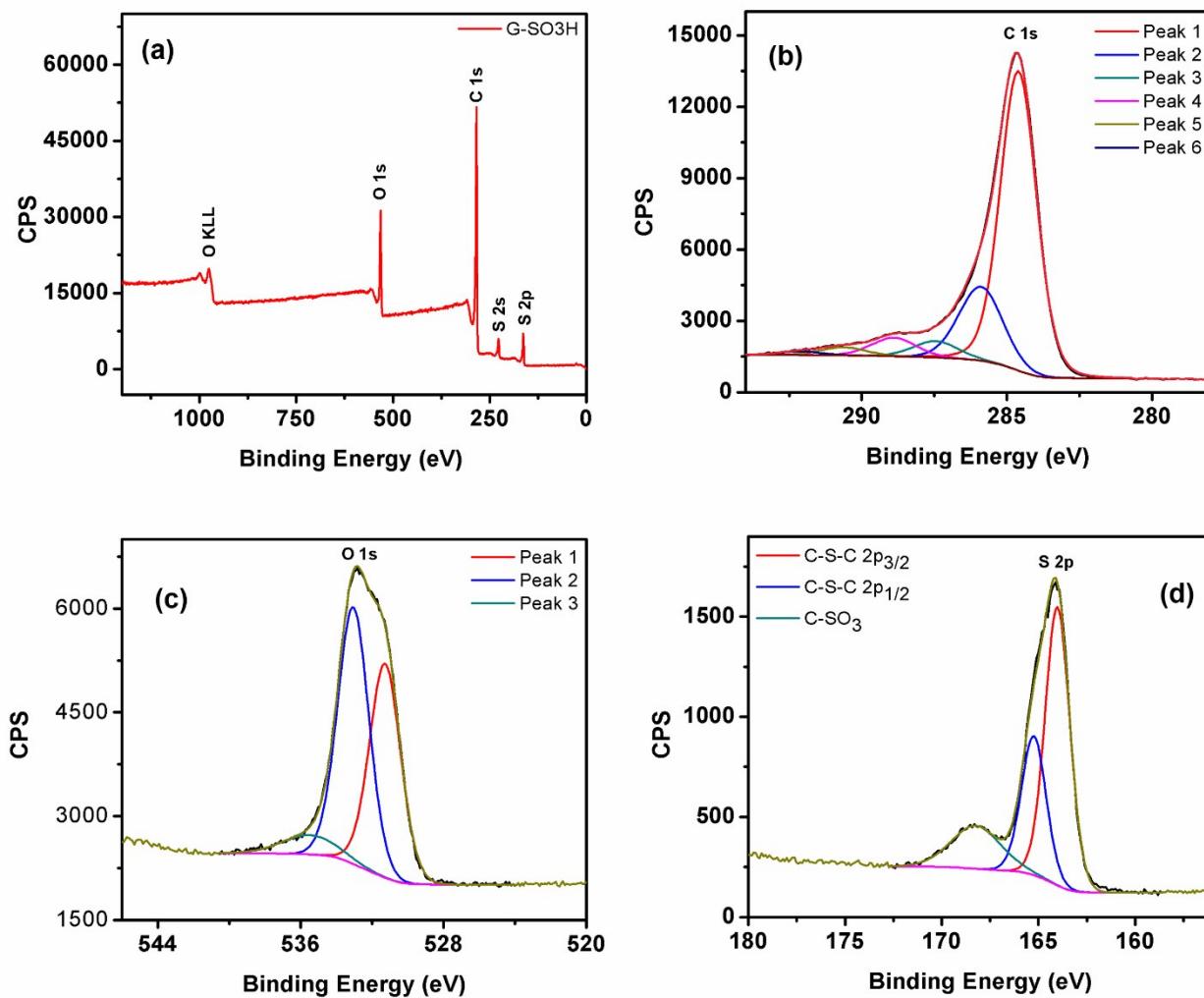


Figure S2. XPS survey spectrum (a) and core-level spectra of C 1s (b), O 1s (c), and S 2p (d) for the G-SO₃H catalyst.

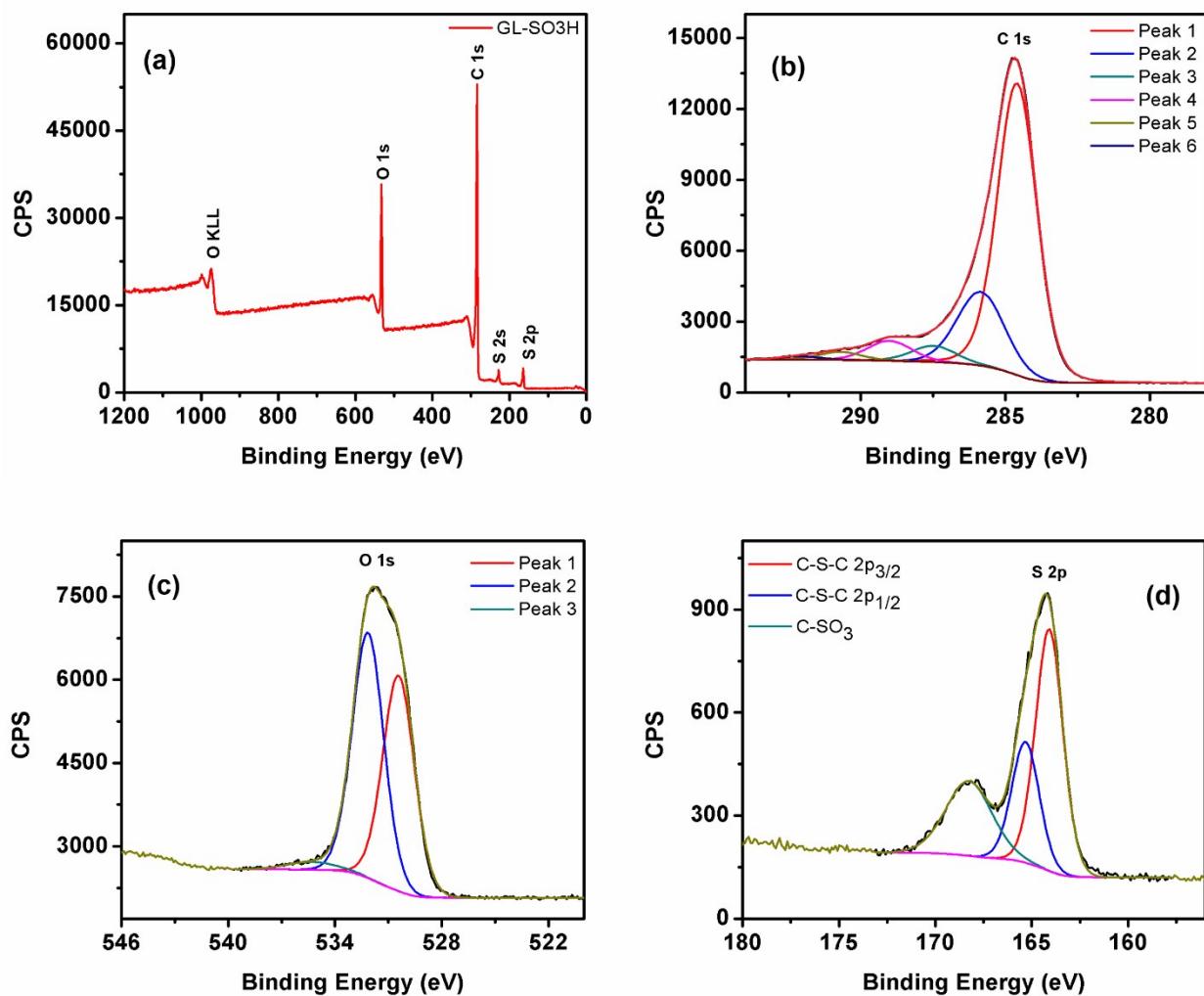


Figure S3. XPS survey spectrum (a) and core-level spectra of C 1s (b), O 1s (c), and S 2p (d) for GL-SO₃H catalyst.

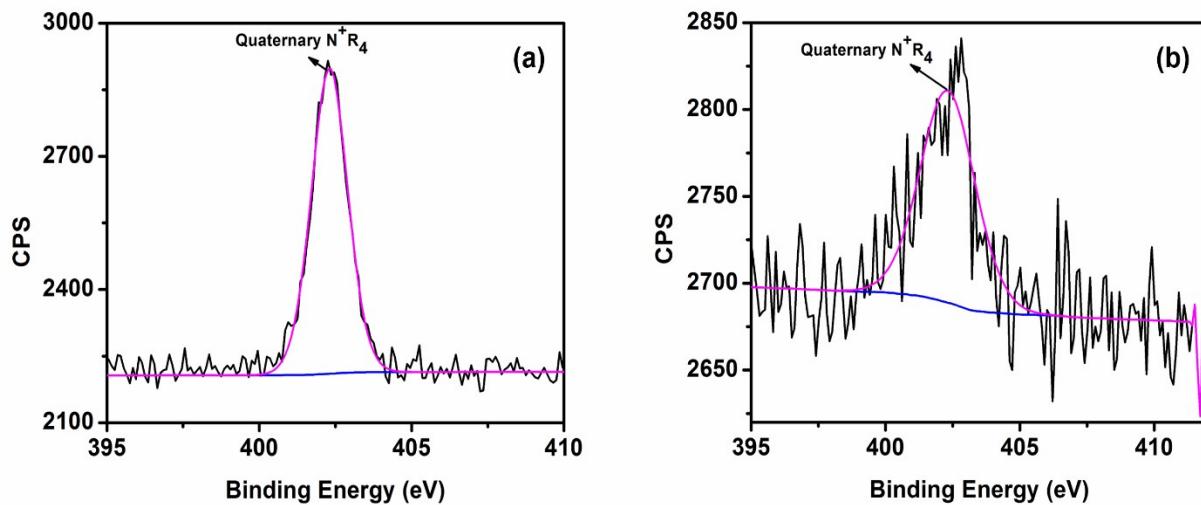


Figure S4. XPS core-level N (1s) spectra of GS-SO₃H (a) and GSR-SO₃H (b) catalysts.

Table S1. CHNS/O analysis (mass %) of the catalysts

Catalyst	N (%)	C (%)	H (%)	S (%)	O (%)
G-SO₃H	0.0	67.0	3.7	6.1	23.3
GL-SO₃H	0.0	65.1	3.7	7.4	19.3
GS-SO₃H	1.2	64.1	6.1	6.7	17.3

Table S2. Atomic concentration of the elements from the high-resolution core-level spectra.

Catalyst	N (%)	C (%)	S (%)	O (%)
G-SO₃H	-	79.1	4.2	16.7
GL-SO₃H	-	79.2	2.4	18.4
GS-SO₃H	1.6	79.8	2.3	16.3
GSR-SO₃H	0.4	76.8	4.6	18.2

Section S3: Effect of different reaction parameters

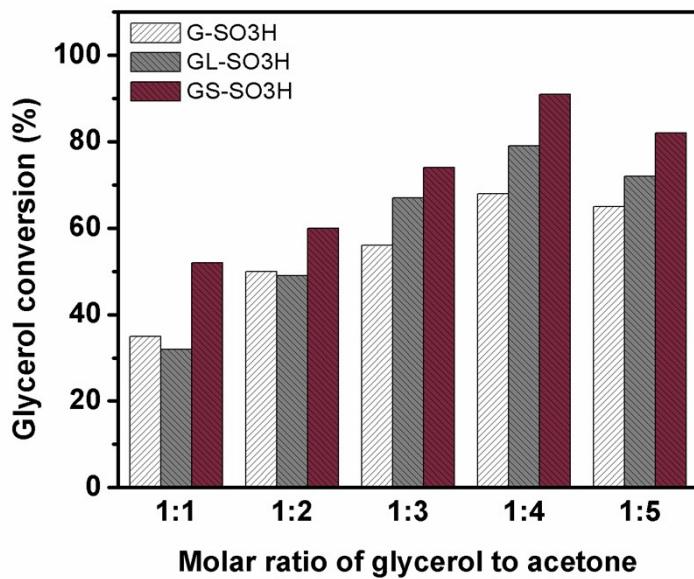


Figure S5. Acetalization of glycerol under various glycerol to acetone mole ratio over different carbon-based catalysts. Reaction condition: Room temperature, reaction time: 4 h, catalyst amount= 5 wt. % (w.r.t glycerol)

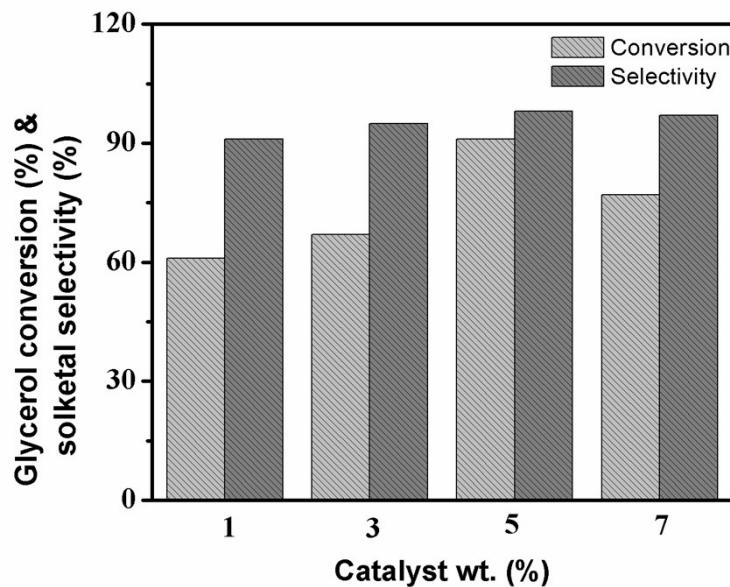
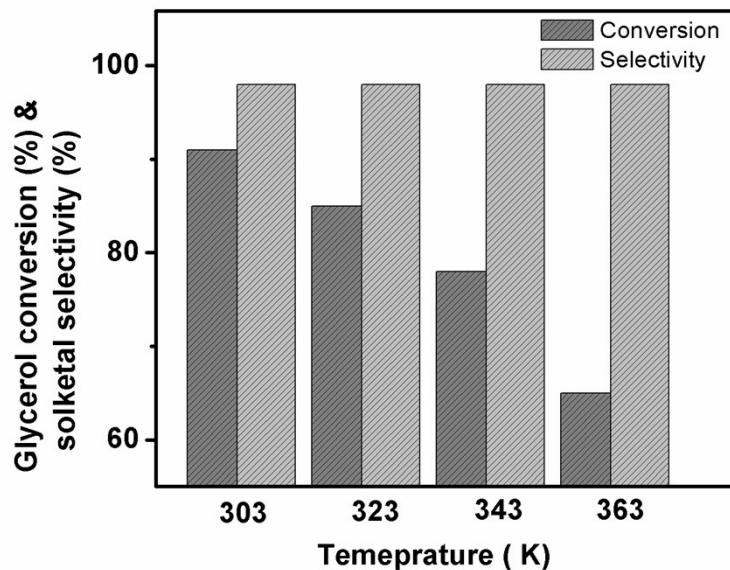


Figure S6. Variation of catalyst wt. (%) for glycerol acetalization over GS-SO₃H catalyst.

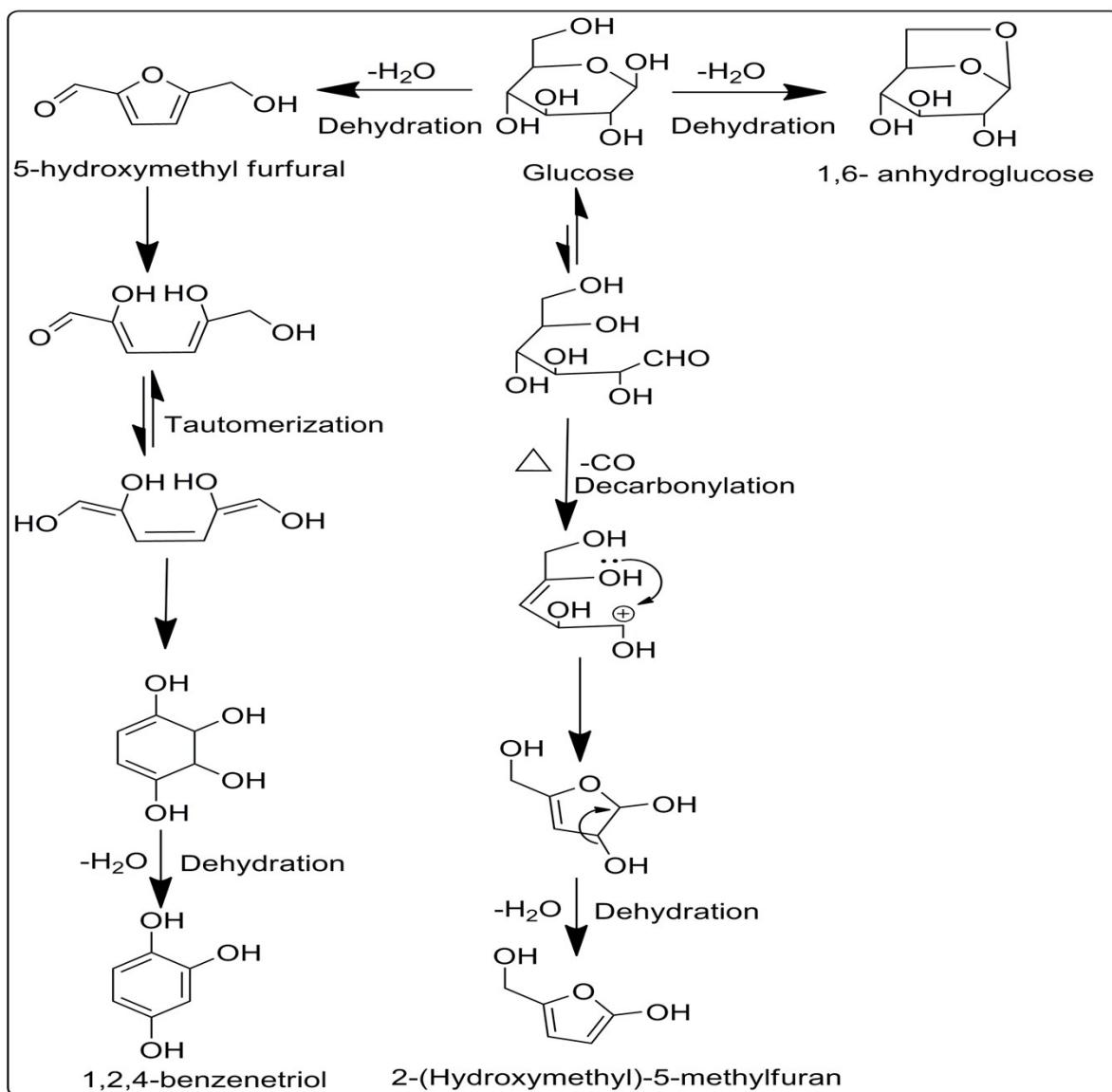


Reaction condition: glycerol: acetone =1:4, room temperature, reaction time = 4 h.

Figure S7. Variation reaction temperature for glycerol acetalization over GS-SO₃H catalyst.

Reaction condition: glycerol: acetone =1:4, room temperature, reaction time = 4 h, catalyst amount= 5 wt. % (w.r.t glycerol).

Section S4: Macromolecules formation mechanism



Scheme S1. Dehydration, decarbonylation, and aromatization of glucose under hydrothermal condition.

Section S5: Characterization of GSR-SO₃H (used) catalyst

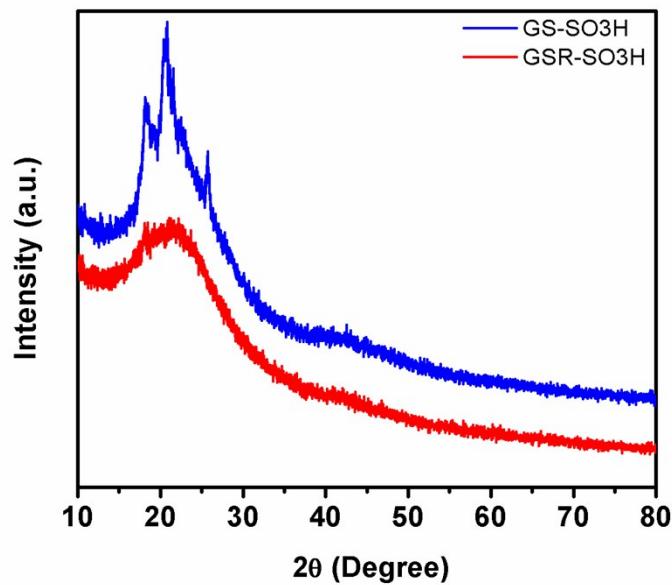


Figure S8. XRD analysis of GS-SO₃H and GSR-SO₃H catalysts.

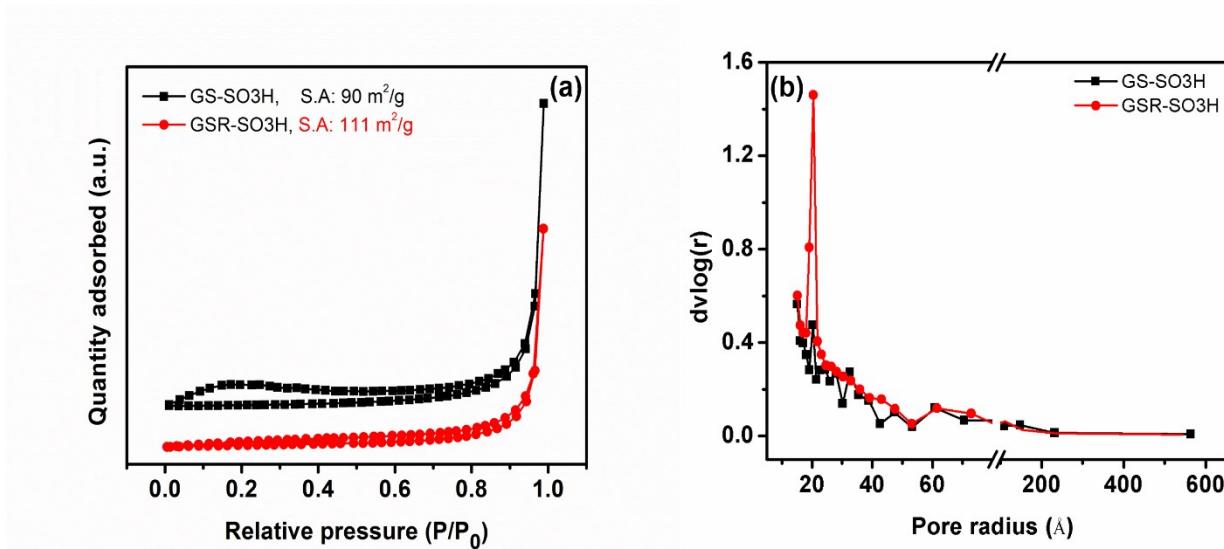


Figure S9. N₂ adsorption/desorption analysis (a) and BJH pore size distribution plot (b) of GS-SO₃H and GSR-SO₃H catalysts.

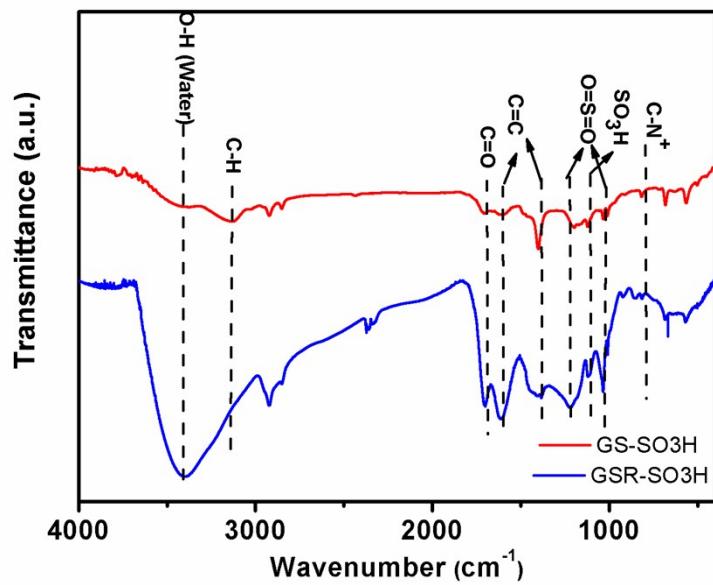


Figure S10. FTIR spectra of GS-SO₃H and GSR -SO₃H catalysis.

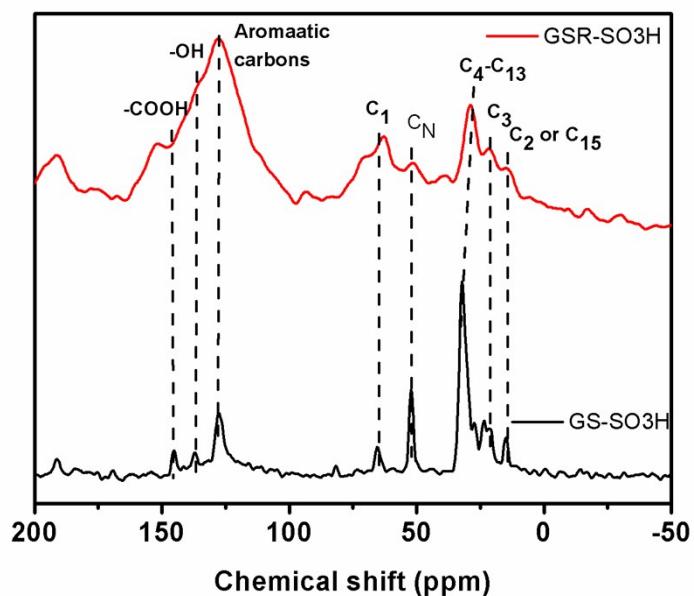


Figure S11. CP MAS ^{13}C -NMR spectra of GS-SO₃H and GSR-SO₃H catalysts.

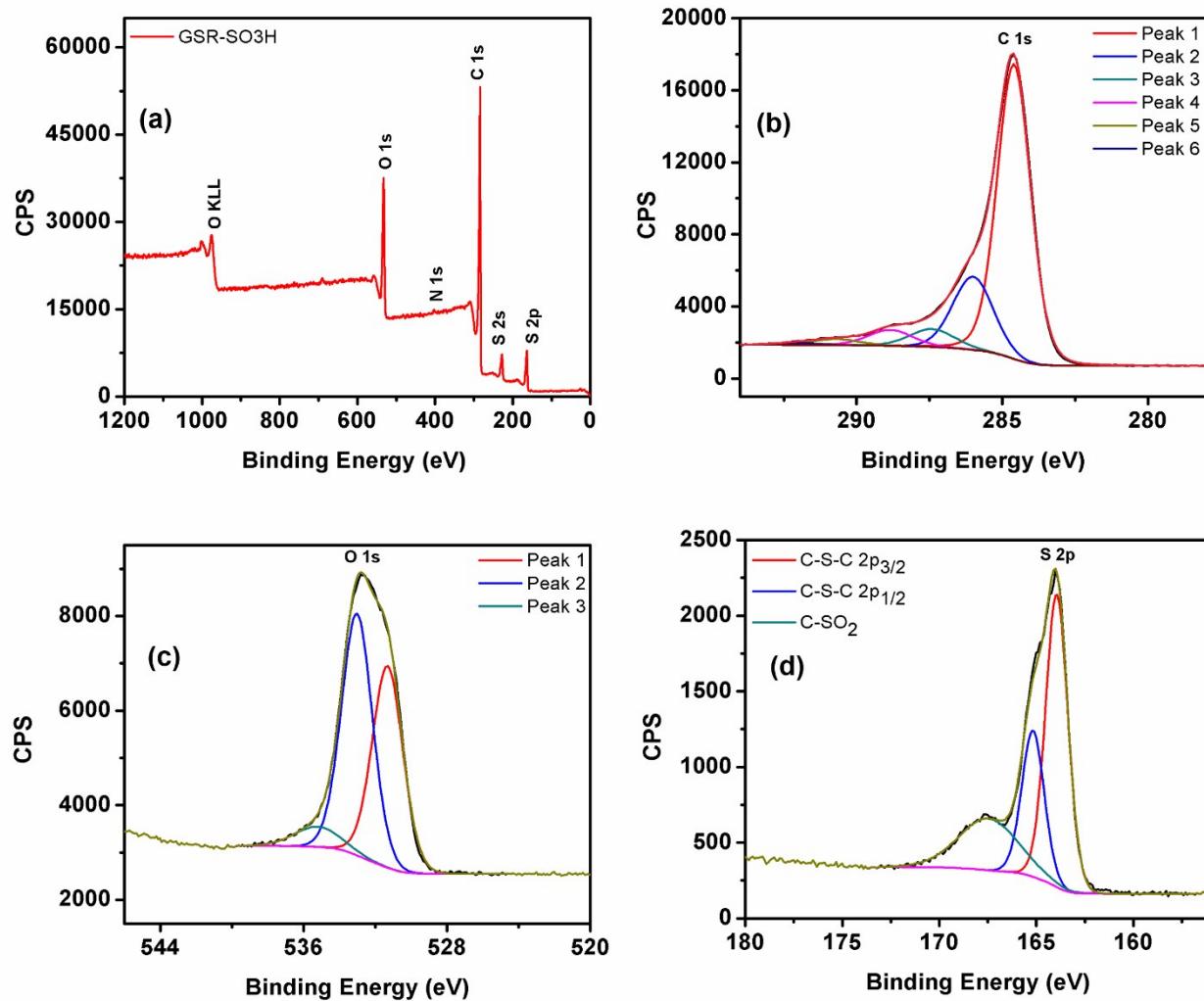


Figure S12. XPS survey spectrum (a) and core-level spectra of C 1s (b), O 1s (c), and S 2p (d) for GSR-SO₃H catalyst.

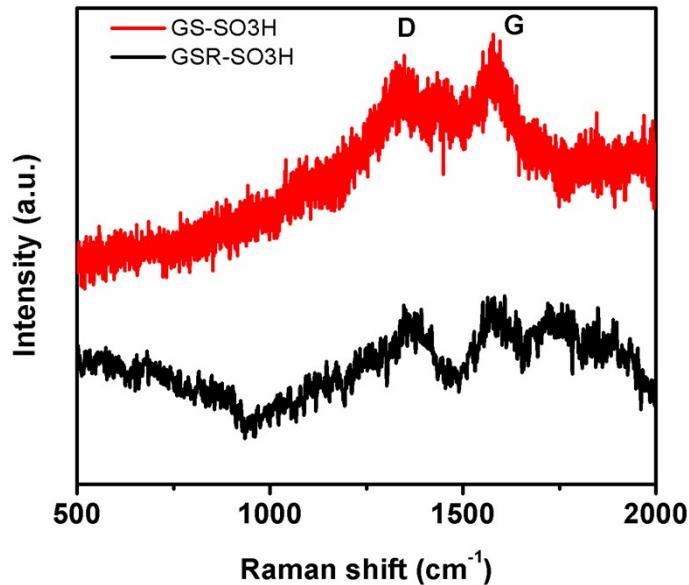


Figure S13. Raman spectra of GS-SO₃H and GSR -SO₃H catalysis.

Section S6: Calculation of atomic percentage to mmol g⁻¹

Atomic % of S = the total number of S atoms out of 100

$$= \frac{\text{total number of } S \text{ atoms}}{N_A} \quad \text{moles per 100 atom}$$

These 100 atoms now contain carbon, oxygen, sulfur, and nitrogen

$$\begin{aligned} & \frac{(\text{total number of } C \text{ atoms} \times M_C) + (\text{total number of } O \text{ atoms} \times M_O) + \\ & \quad (\text{total number of } S \text{ atoms} \times M_S) + (\text{total number of } N \text{ atoms} \times M_N)}{N_A} \\ \text{Total weight} &= g \\ S \text{ content} &= \frac{\text{total number of } S \text{ atoms}}{\text{Total weight} \times N_A} \quad \text{mmol g}^{-1} \end{aligned}$$