Electronic Supplementary Material (ESI) for RSC Advances.

Supplementary Information

Rib shaped carbon catalyst derived from *Zea mays L.* cob for ketalization of glycerol

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Section 1 Experimental Section

a. Catalyst preparation

The catalyst was prepared using agro-waste, i.e. corn cob. The received corn cob was dried in the open air 2-3 days to remove the excess moisture. The dried biomass was ground into small pieces of 75 mesh sizes. The ground corn cob was calcined in the muffle furnace using the prepared crucible (vol~ 0.01 m^3) to provide the inert atmosphere during calcination(Figure 1). The calcination temperature was kept at 500 °C and done for 1-2 hours. The calcined biomass was then activated using NaOH pellets as an activating agent with a molar ratio of 1:3 (Biomass: NaOH). The activated carbon, thus produced is again heated in a muffle furnace at a temperature of around 450-500 °C. After heating the activated carbon, we got a semi-liquid which was then filtered using Whatman filter paper while washing it continuously with distilled water to remove the excess alkali present in it. The collected filtrate was then heated in an oven 80-100 °C for 8-10 hours to remove the moisture. The final product was then stored in an airtight bottle which was our desired activated carbon catalyst prepared using corn cob for the glycerol conversion and was labelled as AC-CC.

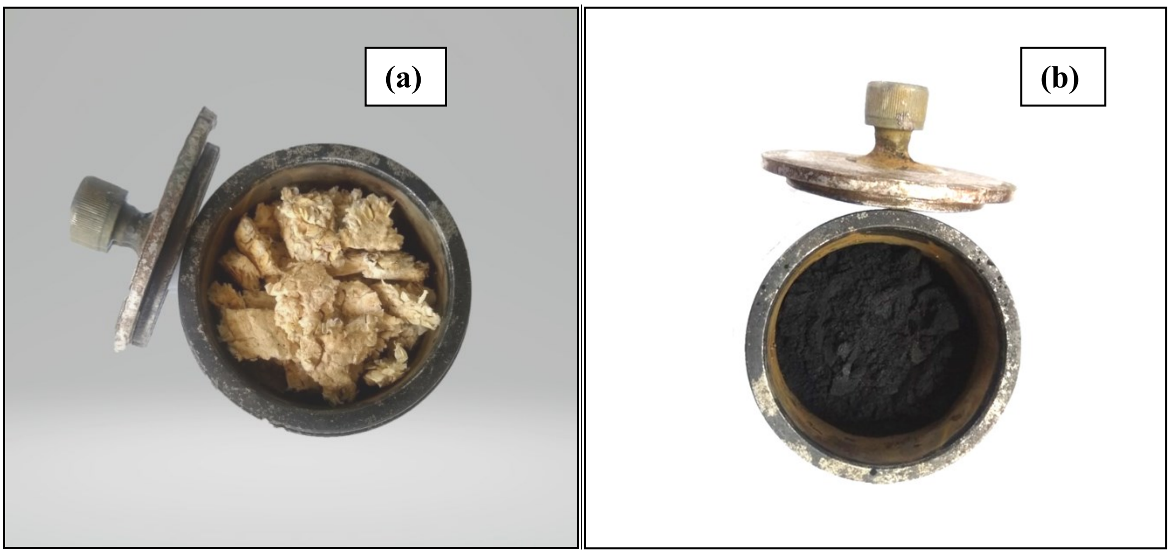

Figure 1: Crucible used for the carbonisation of the biomass (a) with raw material, (b) with derived powdered catalyst

b. Ketalization reaction

The reaction was carried out in a three-round neck flask of 250 ml with varying reaction parameters such as the molar ratio (glycerol: acetone: methanol), reaction temperature, reaction time, catalyst loading (wt. %). The flask is then kept in a beaker of 1000 ml containing water which acts as a water bath (Figure 2). This beaker is then placed on a magnetic plate with a stirrer and temperature controller to control the temperature and uniform mixing of the reaction mixture during the reaction. Two necks of the flask were blocked by a flask glass stopper and in one reflux and condense is also attached to the flask with the help of the burette stand, to maintain the uniformity of the reaction mixture as some of the components are volatile in nature which may evaporate. The reaction was then preceded at 600 rpm. Total 30 sets of reactions are performed with varying molar ratio from 1:2:2 to 1:8:8, temperature – 50 – 150 °C, time – 1-3 hours, catalyst amount – 1 – 5 wt. %. Thus, we have 30 samples to analyze each for one reaction. Each sample collected after the completion of the reaction was then centrifuged using Centrifuge 5430 R to remove the traces of the catalyst present in it. The centrifuged samples were then diluted to 100 times and were analyzed using HPLC (Agilent Technologies model 1260 Infinity) with the Hyplex-H column taking 5mM H_2SO_4 as the solvent.

Fig. 2 Batch reactor set up for the ketalization reaction

Section -II

Analysis section

The samples are collected after the completion of each reaction. They are then centrifuged using Centrifuge 5430 R to remove the traces of the catalyst present in it. The centrifuged samples were then diluted to 100 times and were analyzed using HPLC (Agilent Technologies model 1260 Infinity) with the Hyplex-H column taking 5mM H_2SO_4 as the solvent.

$$\text{Glycerol Conversion (\%)} = \left[\frac{Peak\ area\ of\ product}{Peak\ area\ of\ reactant + Peak\ area\ of\ product}\right]*100$$

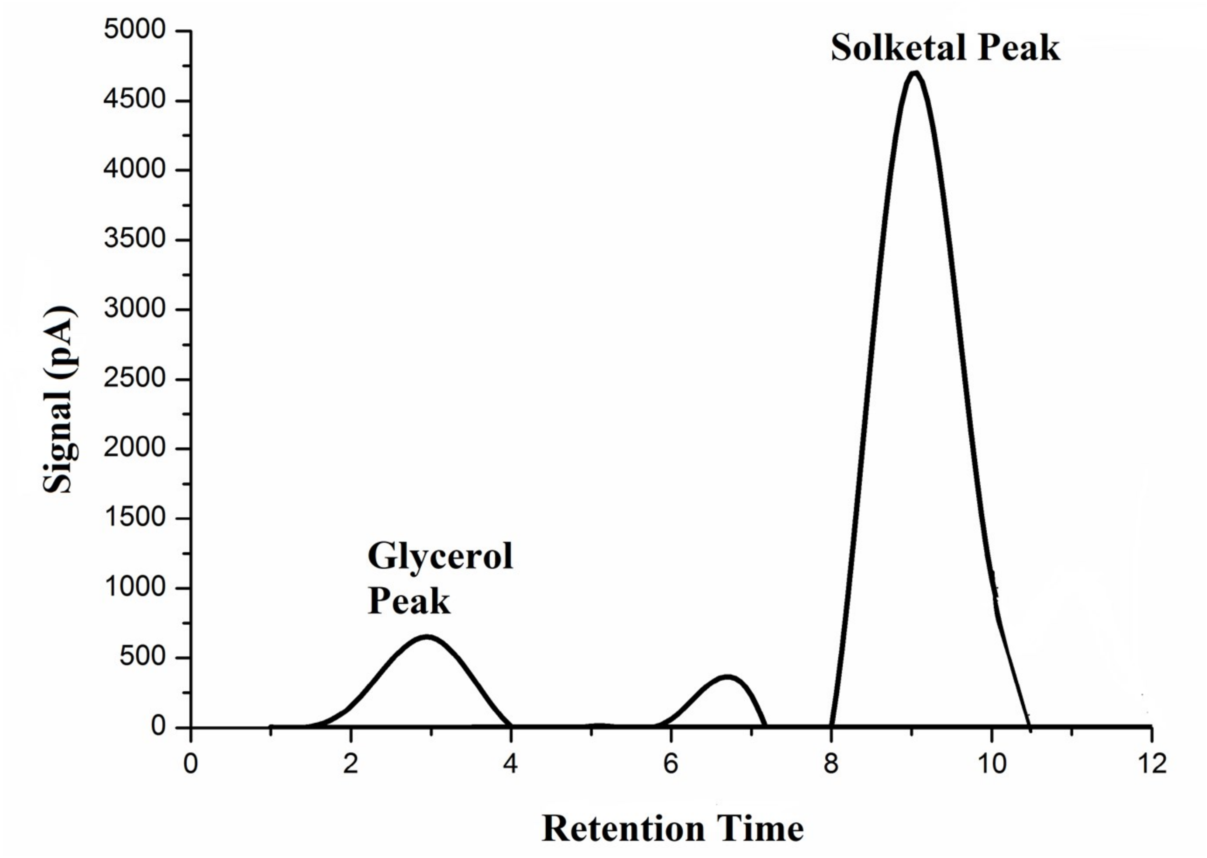

Figure 3: HPLC profile of one of the sample analysed

The conversion of the glycerol to solketal has been estimated from both angle, peak area and peak intensity of the HPLC profile.