Electronic Supplementary Material (ESI) for Sustainable Energy & Fuels.

Supporting Information

The Highly Selective Synthesis of 5-Methy Vanillin from the By-product in Vanilla Industry and The Scent Influence for Vanillin

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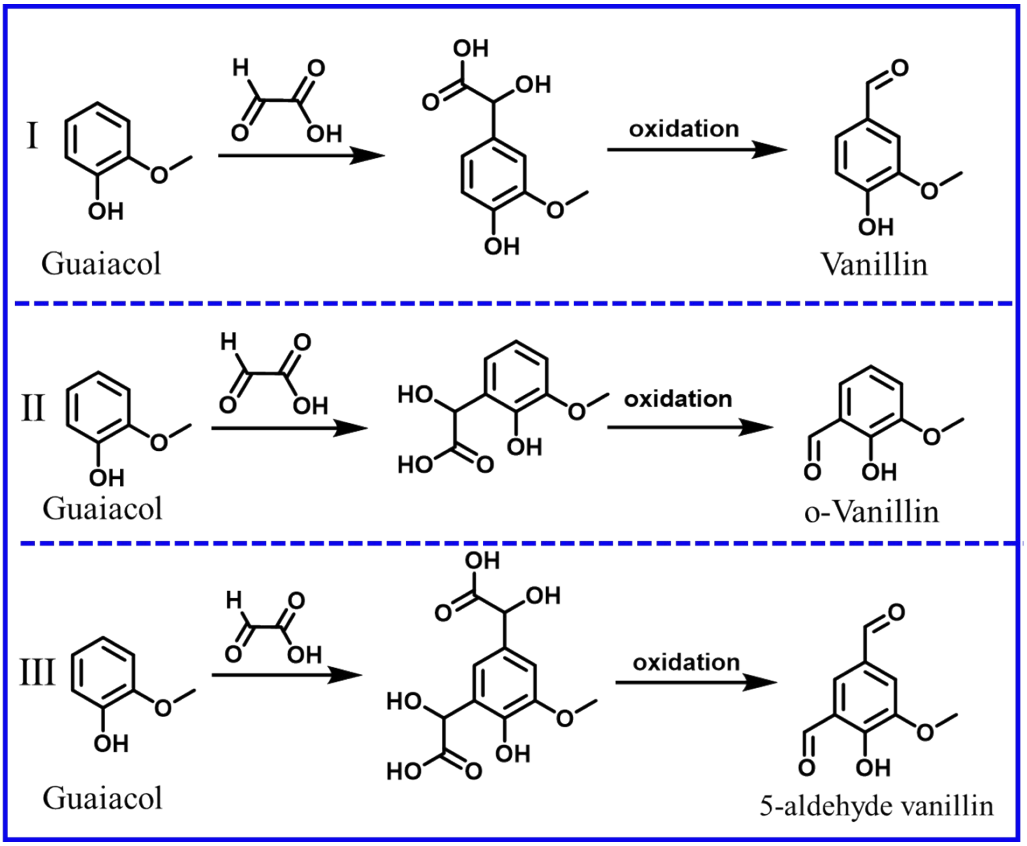
1 Preparation and characterization of Pd/CSC

1.1Preparation of Catalyst Pd/CSC

To remove impurities and modify surface functional groups, the CSC(Coconut shell carbon) (10.0 g) was refluxed at 90 °C in HNO_3(100 mL) for 6 hours. After that, the mixture was cooled to room temperature and then filtered and washed repeatedly using distilled water for 3 times and dried in vacuum. Finally, the obtained CSC was dried at 120 °C for 12 hours. The supported Pd catalysts were prepared by incipient-wetness impregnation of CSC with aqueous solutions of $Pd(NO_3)_2$ respectively. After impregnation, the catalysts were dried in air at 110 °C for 3 hours and finally reduced in H_2 at 400° C for 3 hours.

1.2 Characterization of the Prepared Pd/CSC Composite Catalyst

X-ray data on suitable single crystals were collected at 293 K with a Focus D8 (Bruker, Germany) with Cu Kα radiation (λ = 0.1542 nm). The composition and structure of these catalyst samples were investigated using a Raman spectrometer (DXR, Thermo Fisher Scientific, America). The X-ray photoelectron spectroscopy (XPS) experiments were performed on a Thermo Escalab 250Xi system using Al Kα radiation (hν = 1486.6 eV). The Brunauer-Emmett-Teller (BET) specific surface areas of typical products were obtained at 77 K in a Micromeritics ASAP 2020 system. The morphology of the as-prepared catalyst was characterized by field-emission scanning electron microscopy (S-3400N, Hitachi, Japan).



Scheme S1 The preparation method of Vanillin form guaiacol.

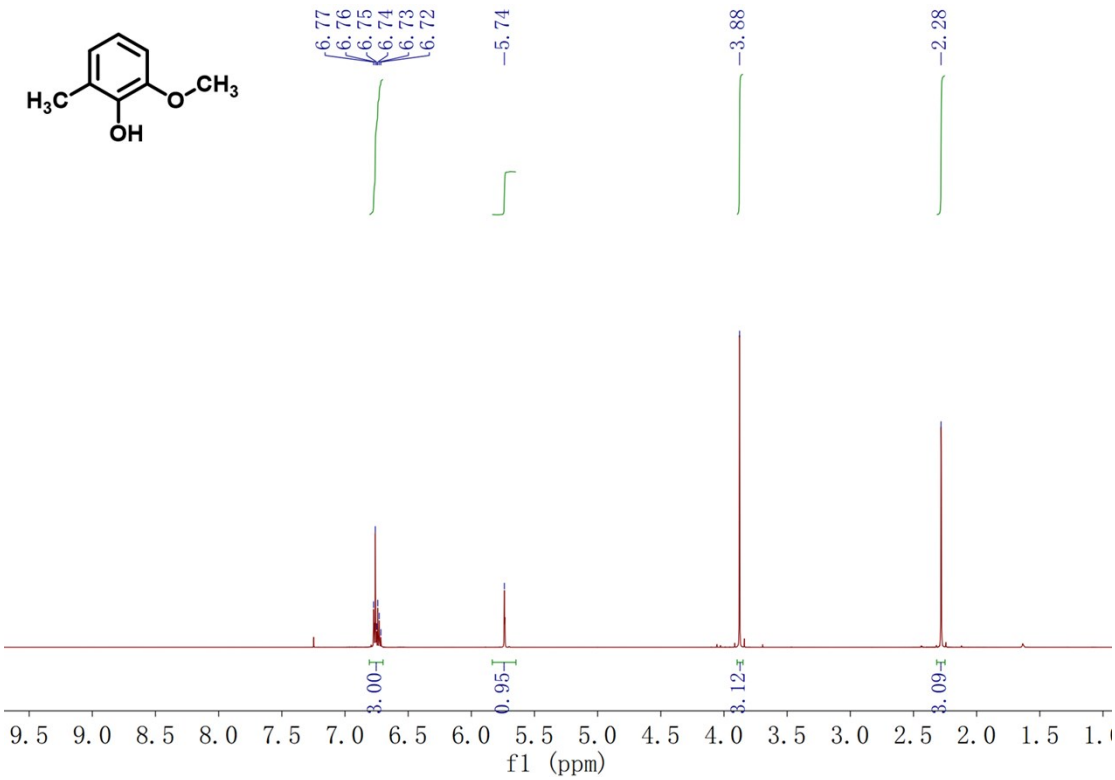


Fig.S1 The [1]HNMR of M-Guaiacol

[1]H NMR (600 MHz, $CDCl_3$, Me_4Si) 6.77-6.73 (3H, m), 5.74 (1H, s), 3.88 (3H, s), 2.28 (3H, s).

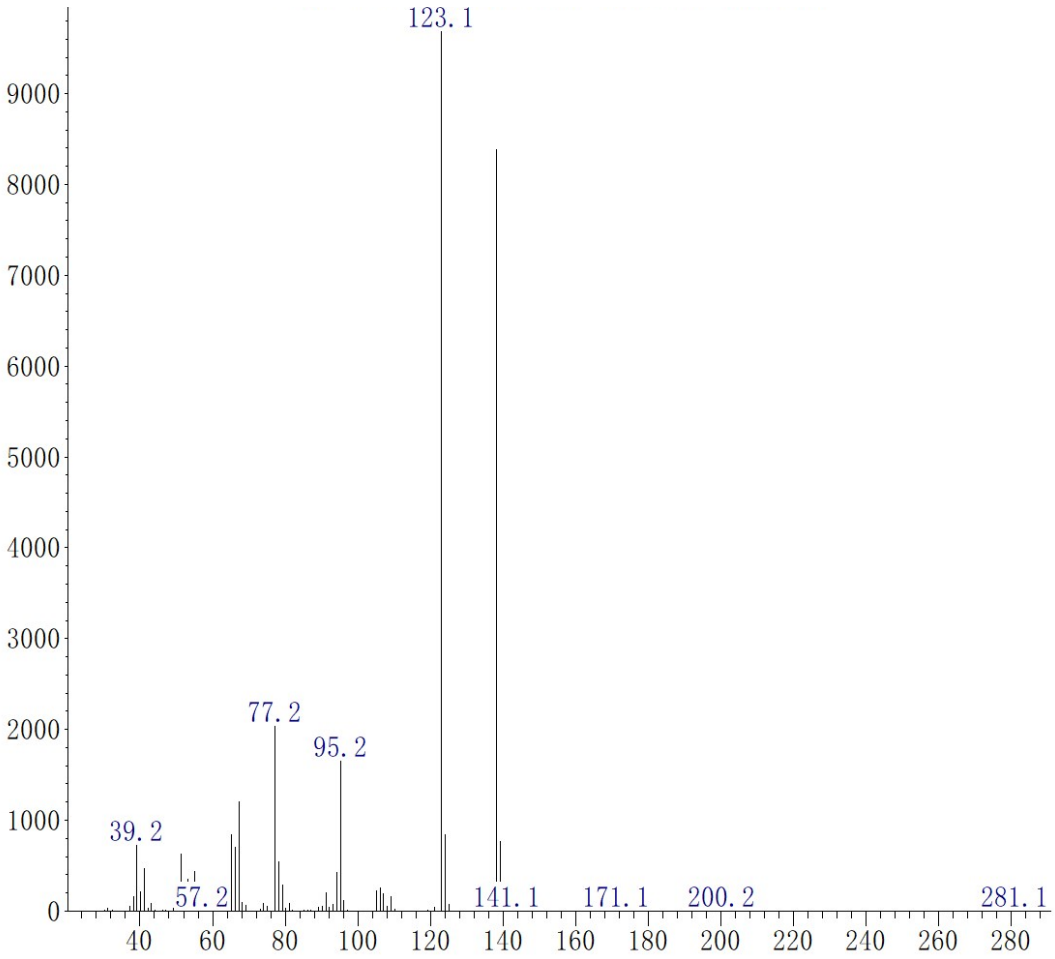


Fig.S2 Gas chromatography - mass spectrometry(GC-MS) of M-Guaiacol.

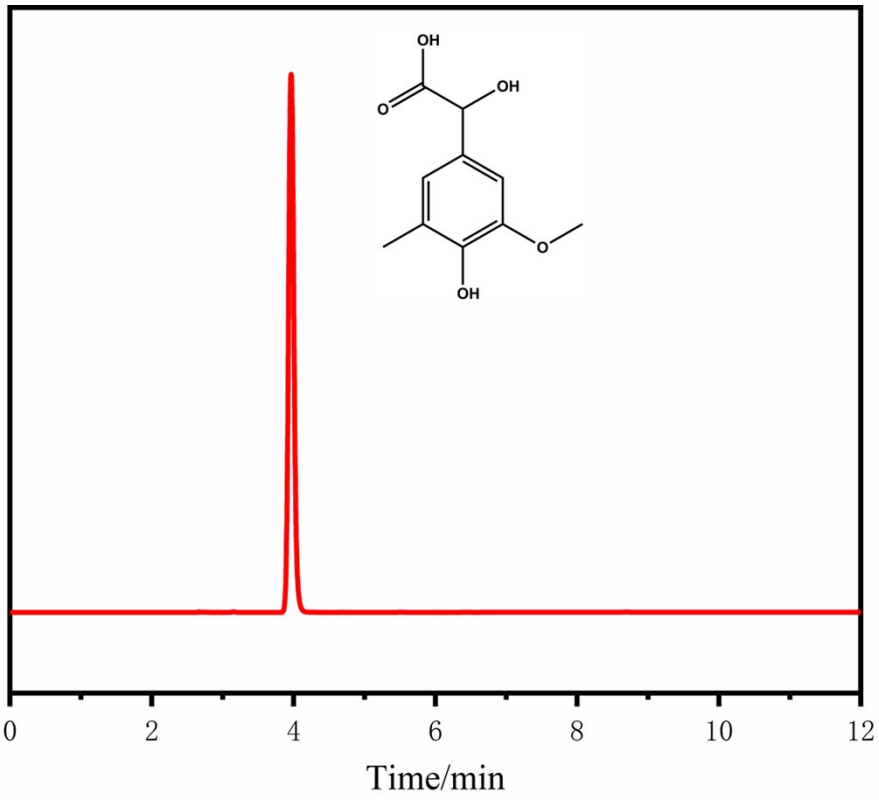


Fig.S3 Liquid phase spectrogram of 5-MVMA.

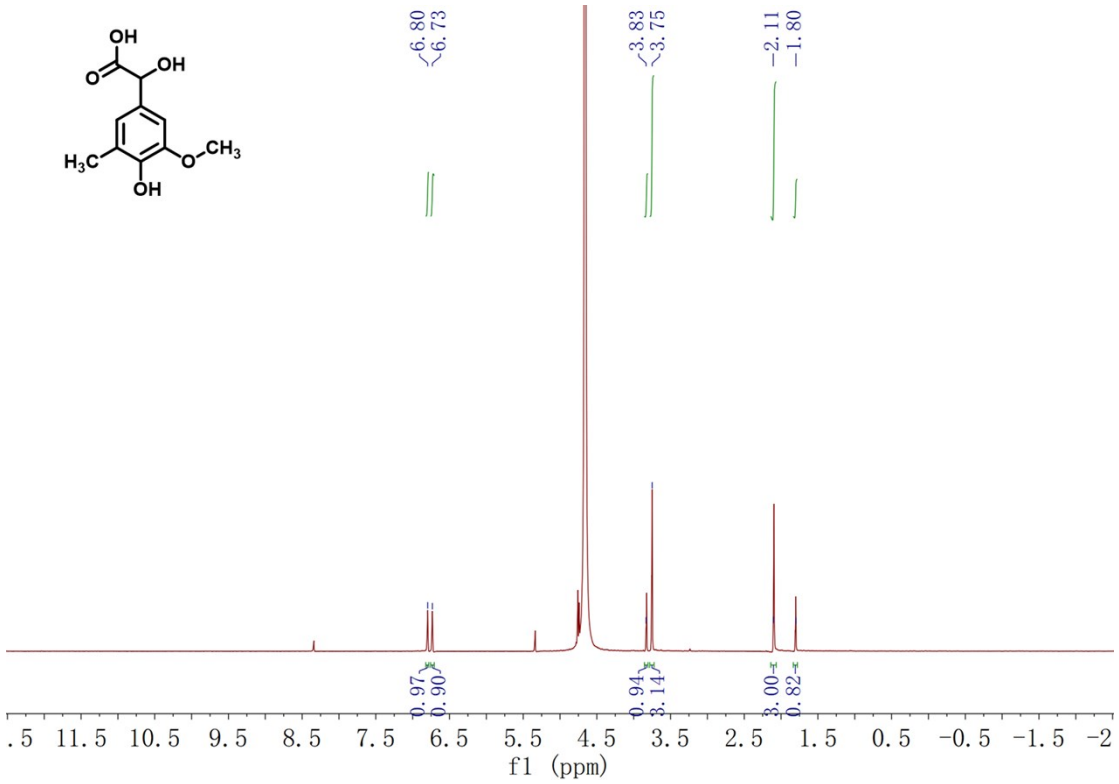


Fig.S4 The [1]HNMR of 5-MVMA

^{1}H NMR (600 MHz, D_2O, Me_4Si) 6.80 (1H, s), 6.73 (1H, s), 3.83 (1H, s), 3.75 (3H, s), 2.11 (3H, s), 1.80 (1H, s).

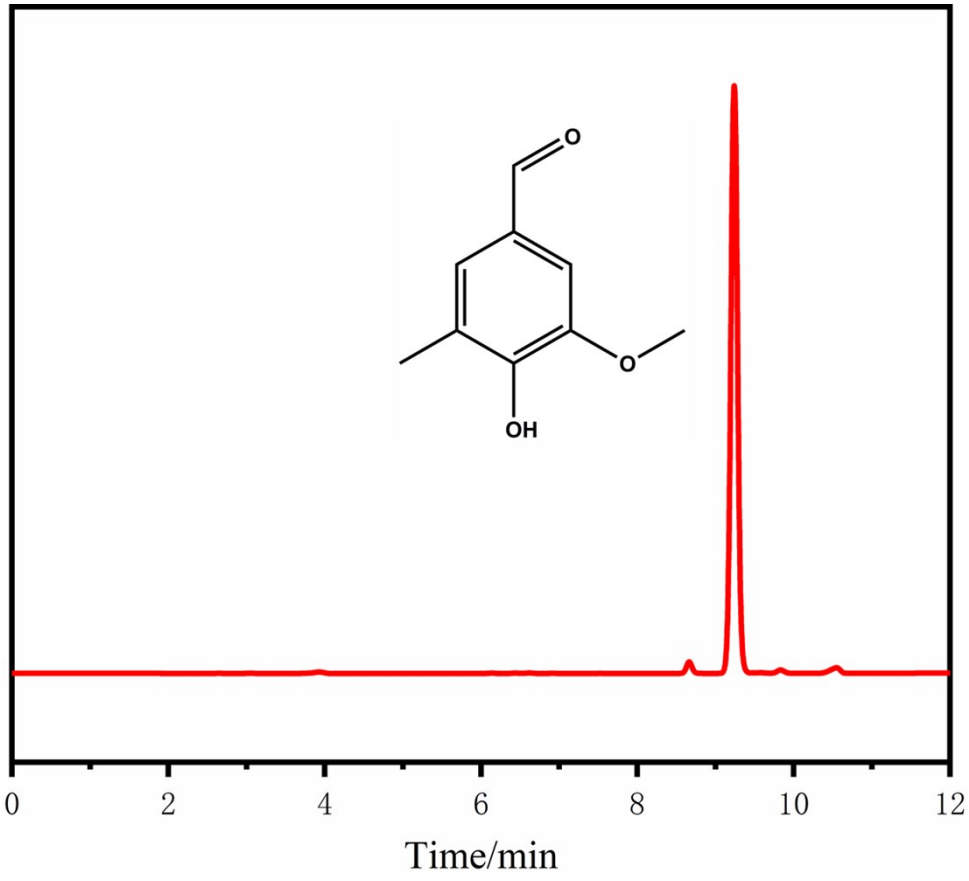


Fig.S5 Liquid phase spectrogram of M-Vanillin.

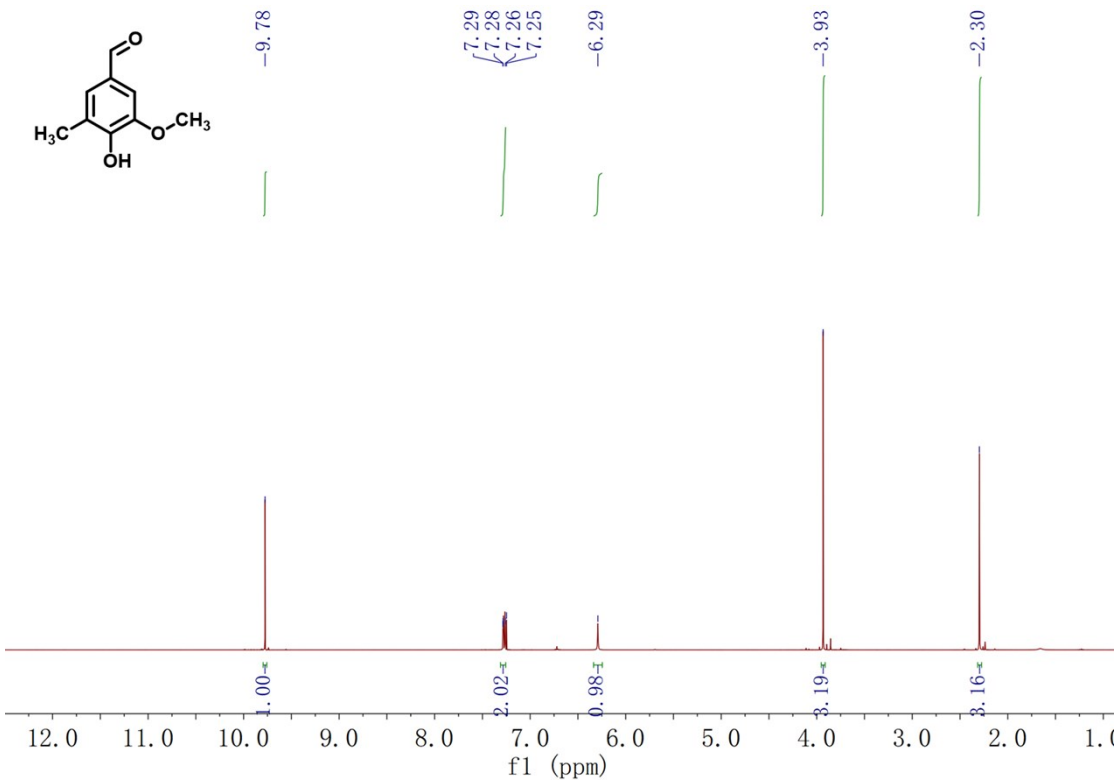


Fig.S6 The ^{1}HNMR of M-Vanillin

^{1}H NMR (600 MHz, $CDCl_3$, Me_4Si) 9.79 (1H, s), 7.29-7.26 (2H, m), 6.29 (1H, s), 3.93 (3H, s), 2.30 (3H, s).

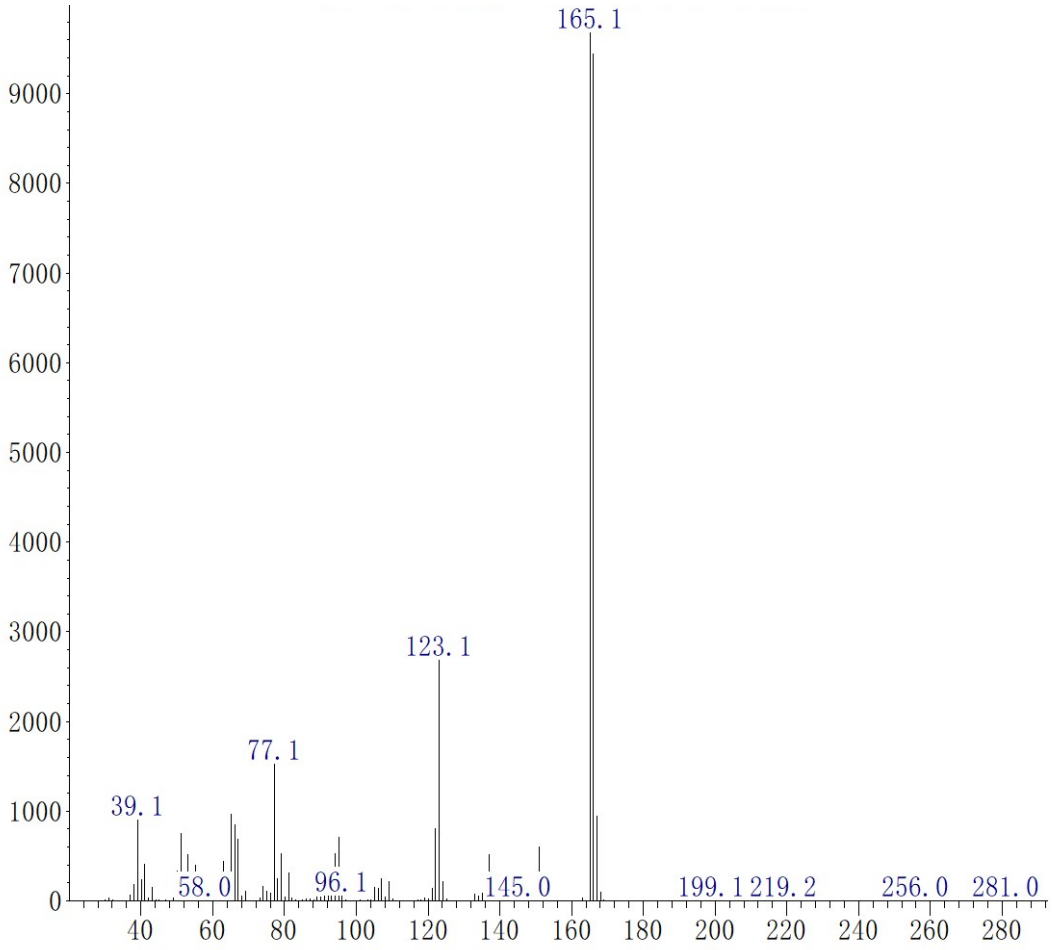


Fig.S7 Gas chromatography - mass spectrometry (GC-MS) of M-Vanillin.

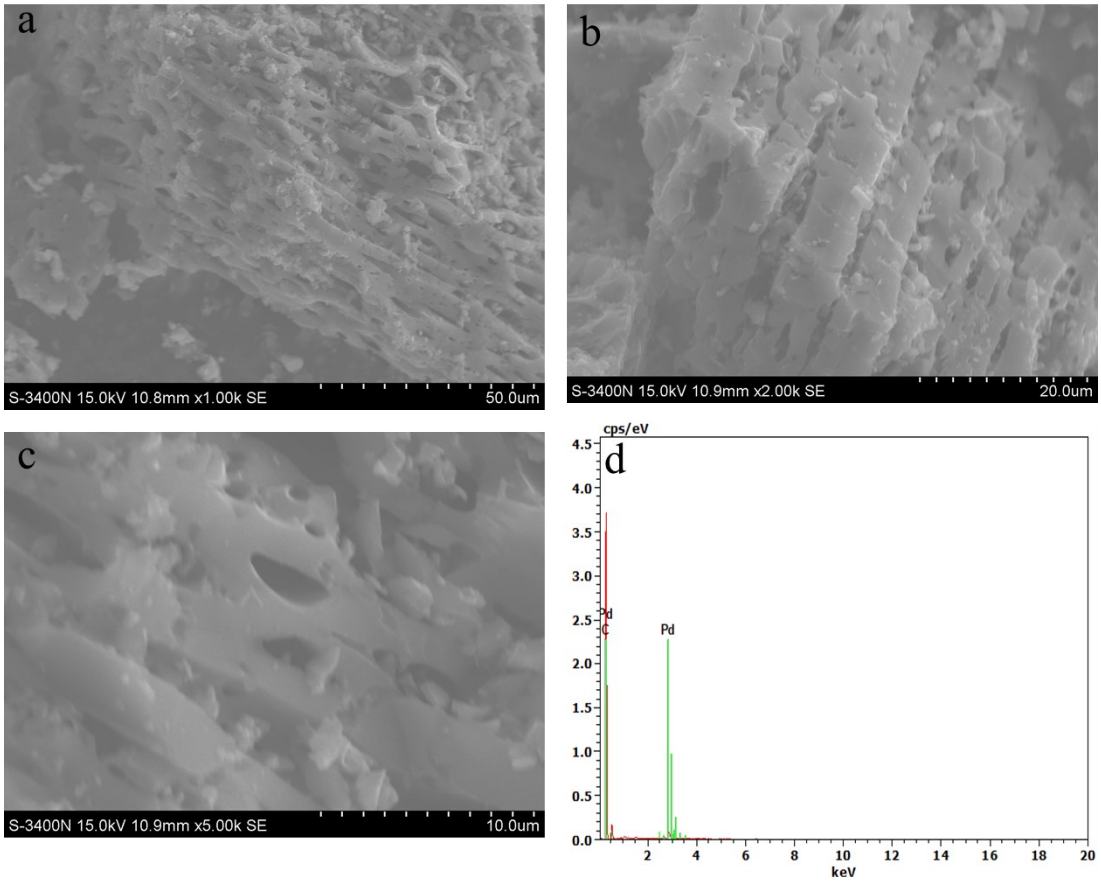


Fig.S8 (a~c) Field Emission Scanning Electron Microscope (SEM) sketch of Pd/CSC, (d) Energy dispersive spectroscopy (EDS) sketch of Pd/CSC.