

reaction time a sample was removed from the reactor by cooling to 40° C., venting the oxygen to atmospheric pressure and withdrawing the sample through the top valve using a syringe and an 18 gauge needle. The sample was analyzed utilizing liquid chromatography and indicated that 80% of original HMF had reacted with approximately 68% conversion to DFF and 32% conversion to FFCA. The autoclave was charged to 150 psi with oxygen and was again heated to 100° C. for an additional 17 hours. The reactor was then cooled and vented and another sample removed. After a total of 23 hours reaction the HMF was completely depleted. Liquid chromatography revealed an absence of detectable DFF and FFCA. The primary product revealed utilizing liquid chromatography analysis was FDCA indicating complete oxidation of HMF. The only other product detected was levulinic acid, which resulted from the hydrolysis of HMF.

Example 3

Preparation of DFF from HMF

1.155 grams of HMF was dissolved in 50 mL of methylene chloride. 7.0606 grams of activated MnO₂ was added to the solution and the mixture was heated to reflux for 8 hours. The MnO₂ was removed from the reaction mixture by filtration and the solids were washed with additional solvent. The solvent was removed to produce and off-white solid. Liquid chromatography analysis of the solid indicated 80% DFF and 20% un-reacted HMF. A trace amount of FDCA was observed utilizing UV detection. The solid was dissolved in hot water and was subsequently cooled to precipitate DFF having a 98.5% purity. Selectivity of the oxidation reaction to DFF was substantially 100%.

Example 4

Preparation of 5% Pt on a ZrO₂ Support

Extrudated ZrO₂ received from Engelhard was calcined at 700° C. for 2 hours. The calcined ZrO₂ was crushed and sieved to 40-80 mesh size. 10.6318 grams of the crushed ZrO₂ was mixed at room temperature with 0.7593 grams of platinum(II) acetylacetonate in 50 mL flask. The flask was then mounted on a rotary evaporator and evacuated by a vacuum pump to reach 10 mmHg. The flask was rotated at 60 rpm for 10 minutes. After a thorough mixing the flask was heated to about 180° C. utilizing a heat gun. During the process the color of the catalyst changed from a light brown color to black. The temperature was then increased to about 240° C. Heating was stopped after approximately 20 minutes. The catalyst was then calcined in air for about 3 hours at 350° C. with a temperature ramp rate of 5° C. per minute.

Activation was carried out by reducing the catalyst in a fixed-bed reactor at 330° C. for 3 hours. The hydrogen flow rate was 40 mL/min. After reduction the reactor was cooled to room temperature under hydrogen and was then purged with helium for 30 minutes. Passivation was conducted by flowing

2% O₂ into the reactor at 40 mL/min overnight. The catalyst was unloaded from the reactor and was transferred to a storage container until use.

In compliance with the statute, the invention has been described in language more or less specific as to structural and methodical features. It is to be understood, however, that the invention is not limited to the specific features shown and described, since the means herein disclosed comprise preferred forms of putting the invention into effect. The invention is, therefore, claimed in any of its forms or modifications within the proper scope of the appended claims appropriately interpreted in accordance with the doctrine of equivalents.

The invention claimed is:

1. A method of oxidizing hydroxymethyl furfural (HMF), comprising:
 - providing a starting material solution into a reactor, the solution having a pH less than or equal to 7, the solution comprising HMF and water;
 - providing at least one of air and O₂ into the reactor; and
 - contacting the starting material with a catalyst comprising Pt, on a support material comprising at least one of Zr, Al, Si, and Ti, the contacting being conducted at a reactor temperature of from about 50° C. to about 200° C., wherein the method selectively produces diformyl furan relative to all other products, intermediates and byproducts.
2. The method of claim 1 wherein the solution comprises acetic acid.
3. The method of claim 1 wherein the catalyst comprises 5% Pt on a SiO₂ support material.
4. The method of claim 1 wherein the support material comprises at least one of ZrO₂, Al₂O₃, SiO₂, and TiO₂.
5. The method of claim 3 wherein the acetic acid is present at a ratio of 40:60 relative to the water.
6. The method of claim 1 wherein air is provided to the reactor.
7. A method of oxidizing hydroxymethyl furfural (HMF), comprising:
 - providing a solution into a reactor, the solution comprising HMF and water;
 - providing at least one of air and O₂ into the reactor; and
 - contacting the starting material with a catalyst comprising Pt and a support material, the support material comprising one or more of ZrO₂, Al₂O₃, SiO₂, and/or TiO₂, the contacting being conducted at a reactor temperature of from about 50° C. to about 200° C., wherein the method selectively produces diformyl furan relative to all other products, intermediates and byproducts.
8. The method of claim 7 wherein the solution has a pH less than or equal to 7.
9. The method of claim 7 wherein air is provided to the reactor.
10. The method of claim 7 wherein the solution consists essentially of HMF, water, and weak acid.

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