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**PhD Dissertation**

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*CONVERSION OF LIGNOCELLULOSE BIOMASS FROM DATE PALM WASTE TO HIGH VALUE CHEMICALS*

by

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Date & Venue

11:00 AM

Wednesday, 14 April 2021

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Abstract

Concerns about depleting nonrenewable resources especially fossil fuels and increasing greenhouse gas emissions are driving many global economies to develop renewable energy sources at a fast rate. In this context, biomass as a sustainable and carbon-neutral resource may provide a solution. Lignocellulose biomass has gained growing interest because of its low cost of production compared to food crops, it solves problems of food and energy security, can grow in various climates and land, and it is CO<sub>2</sub> neutral. Date palm trees produce large amounts of fibrous wastes that are a good source of lignocellulose. There are over 40 million date palm trees in UAE and most of their lignocellulosic wastes (i.e., leaves, surface fibers, bunches, etc.) are currently used for low value products such as compost or electric generation. However, the lignocellulosic components of this waste stream have potential to be used to produce high-value chemicals (HVC). Conversion technologies such as thermochemical, biochemical, physiochemical, and biotechnological are often used to convert lignocellulose biomass into valuable products (fuels, chemicals, etc.). The thermochemical technique uses heat energy and catalysts to break and upgrade biomass polymers into valuable products.

Reductive catalytic fractionation (RCF) under thermochemical conditions were applied to lignocellulose biomass of date palm waste for HVC production and an integrated date palm waste-based biorefinery was proposed which includes date palm waste collection, waste valorization and catalytic conversion to HVC. The advantages of using thermochemical techniques are thoroughly discussed in the literature although the actual application of the technique in industry is still far from being pervasive.

The results described in this dissertation are a collection from six publications and a submitted patent application and have been divided into five chapters. The findings indicate good energy potential with high average total volatile matter and high heating values. The thermal gravimetric analysis (TGA) results revealed the main mass loss trend is in the order of Rachis > Composite > Leaflet > Fiber in a temperature range of 127–398 °C. To elucidate the role of catalysts towards fractionation of lignocellulose biomass waste to valuable organics, we employed transitional metal phosphide (TMPCs) catalysts due to their high reactivity in hydrotreatment reactions. The characterization results revealed high surface area (BET) of 201, 173 and 154 m<sup>2</sup> g<sup>-1</sup> for Ni<sub>2</sub>P/γ-Al<sub>2</sub>O<sub>3</sub>, CoP/γ-Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>P/γ-Al<sub>2</sub>O<sub>3</sub>, respectively. The catalytic performance demonstrated better liquid yields using a commercial NiAl alloy catalyst than with in-house prepared TMPCs catalysts. The liquid yield improved from a marginal 4 % without a catalyst to a substantial 68 % with a catalyst. The total lignin product yield was lower without a catalyst (16 %) than with a catalyst (76 %). The gas chromatography-mass spectrometry (GC-MS) quantification results revealed production of C<sub>5</sub>–C<sub>12</sub> compounds. These include furfural (C<sub>5</sub>), furfural alcohol (C<sub>5</sub>), phenol (C<sub>6</sub>), m-cresol (C<sub>7</sub>), guaiacol (C<sub>7</sub>), methyl benzoate (C<sub>8</sub>), isoeugenol (C<sub>10</sub>), 2-methoxy-4-propylphenol (C<sub>10</sub>), 5-isopropyl-2-methylphenol (C<sub>10</sub>), 2,6-dimethoxy-4,2-propenyl phenol (C<sub>11</sub>) and 3,4-diethyl-2,4-hexadienedioic acid, dimethyl ester (C<sub>12</sub>). At lower temperature below 240 °C regardless of the prolonged reaction time there was low production of C<sub>5</sub>-C<sub>12</sub> compounds (42 %). However, at higher temperatures the same compounds increased exponentially to a total yield of 77 % with reaction time from 2-6 h. The C<sub>7</sub>–C<sub>8</sub> compounds were the largest product fraction. There were substantial differences between the carbon distributions from the commercial alloy catalyst, supported transition metal phosphide catalyst, and catalyst-free processes. The commercial NiAl catalyst showed the highest g converted per g catalyst per hour (7.34 h<sup>-1</sup>) for a given reaction condition. The results of high volatile matter, energy content, low energy barriers across a wide range of temperature, and thermodynamic feasibility associated with the pyrolysis of date palm waste profess it as a viable biomass candidate in the biorefinery. In addition, the results from this study provide a deep understanding of the role of different process parameters, the positive attributes of the direct conversion method and viability of date palm waste as a potential lignocellulose for production of high-value chemicals. The results further showed the potential of NiAl alloy catalyst and TMPCs towards direct production of improved quality valuable oxygenates from natural lignocellulose waste in a low-cost, timely and environmentally safe operation.

**Keywords:** Depolymerization, lignocellulose, direct conversion process, catalysis, high-value chemicals, waste-to-energy, reaction mechanism; date palm waste, Nonisothermal kinetics.