

prove necessary, a gravitational tractor offers a viable method of controllably steering asteroid 99942 Apophis away from an Earth impact.

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GREEN CHEMISTRY

Biodiesel made with sugar catalyst

The production of diesel from vegetable oil calls for an efficient solid catalyst to make the process fully ecologically friendly. Here we describe the preparation of such a catalyst from common, inexpensive sugars. This high-performance catalyst, which consists of stable sulphonated amorphous carbon, is recyclable and its activity markedly exceeds that of other solid acid catalysts tested for 'biodiesel' production.

The esterification of higher fatty acids by liquid acid catalysts such as sulphuric acid (H_2SO_4) is a process commonly used for biodiesel production, but it involves high consumption of energy and the separation of the catalysts from the homogeneous reaction mixtures is costly and chemically wasteful. Recyclable solid acids, such as Nafion[®]^{1–4}, make better catalysts, although they are also expensive and their activity is less than that of liquid acids¹. Sulphonated naphthalene carbonized at 200–250 °C is a solid acid catalyst that has been used successfully for ethyl acetate formation⁵; however, it is a soft material and its aromatic molecules are leached out during liquid-phase reactions above 100 °C or when higher fatty acids are used as surfactants, so its catalytic activity is rapidly lost.

We have devised a strategy to overcome these problems by sulphonating incompletely carbonized natural organic material to prepare a more robust solid catalyst. Incomplete carbonization of natural products such as sugar, starch or cellulose results in a rigid carbon material that is composed of small polycyclic

aromatic carbon sheets in a three-dimensional sp^3 -bonded structure. Sulphonation of this material would be expected to generate a stable solid with a high density of active sites, enabling a high-performance catalyst to be prepared cheaply from naturally occurring molecules.

The scheme we use to sulphonate incompletely carbonized saccharides is shown in Fig. 1. First, D-glucose and sucrose are incompletely carbonized at low temperature to induce pyrolysis and the formation of small polycyclic aromatic carbon rings; sulphonite groups ($-\text{SO}_3\text{H}$) are then introduced by sulphuric acid (see supplementary information). Structural analysis^{6–8} indicates that the prepared samples consist of sheets of amorphous carbon bearing hydroxyl and carboxyl ($-\text{OH}$ and $-\text{COOH}$) groups, as well as high densities of $-\text{SO}_3\text{H}$ groups.

This black powder is insoluble in water, methanol, benzene, hexane, *N,N*-dimethylformamide and oleic acid, even at boiling temperatures. It can be moulded into hard pellets or thin flexible films by heating with 0.5–5.0% by weight of binding polymer; the two forms have comparable stability and catalytic performance. The thin films act as electrically insulating proton conductors whose properties (0.09 siemens cm^{-1} at 50 °C and 100% humidity) are comparable to that of Nafion (0.1 siemens cm^{-1} at 80 °C).

High-grade biodiesel is produced by esterification of the vegetable-oil constituents oleic acid and stearic acid. The activity of our solid sulphonated carbon catalyst in this reaction is

more than half that of a liquid sulphuric acid catalyst and much higher than can be achieved by conventional solid acid catalysts (see supplementary information). There was no loss of activity or leaching of $-\text{SO}_3\text{H}$ during the process, even for samples subjected to repeated reactions at 80–180 °C after having been recovered by simple decantation. The activity is double that of a carbonized sulphonated naphthalene catalyst tested previously⁵, which decreased rapidly on recycling at 80 °C.

Carbon catalysts identical to those described here have also been successfully produced from carbonized starch and cellulose (results not shown). Saccharide molecules may therefore be generally suitable for preparing these catalysts, which can be used as a replacement for liquid sulphuric acid in esterification reactions. In addition to biodiesel production, such environmentally benign alternative catalysts should find application in a wide range of other acid-catalysed reactions.

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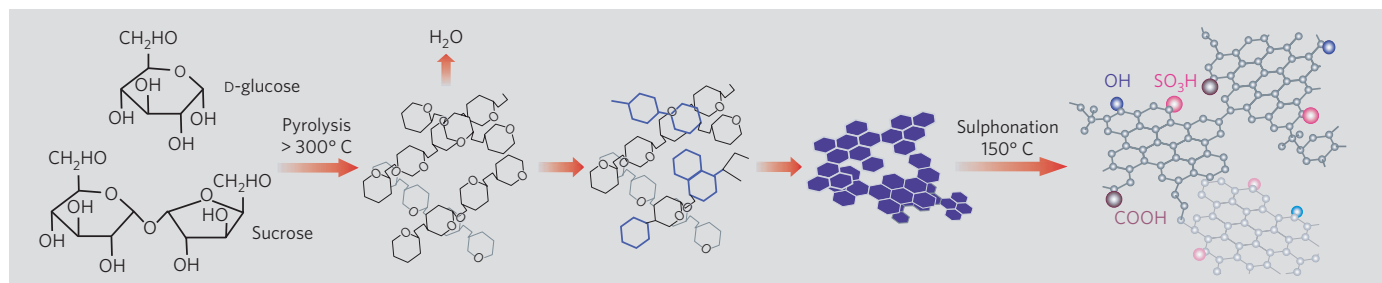


Figure 1 | Preparation from sucrose and D-glucose of a solid catalyst suitable for biological diesel production. Pyrolysis of the sugars causes their incomplete carbonization (middle; outlined in blue) and formation into polycyclic aromatic carbon sheets; sulphuric acid (concentrated or fuming) is used to sulphonate the aromatic rings to produce the catalyst. For details of methods, see supplementary information.