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Synthesis of Solid Superacid Catalyst with Acid Strength of $H_9 < -16.04^*$ <b>11 h 22</b> Summary A solid superacid catalyst with an acid strength of $H_0 \wedge -16-04$ , which was active for reactions of propane and butane, was obtained by	Tổng hợp chất xúc tác siêu axit rắn có độ axit..... Tóm tắt Trong công trình này, chúng tôi tiến hành điều chế chất xúc tác siêu axit rắn có độ axit là....., (chất này kích hoạt các phản ứng propane
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exposing  $Zr(OH)_4$ , prepared by the hydrolyses of  $ZrOCl_2$  and  $ZrO(NO_3)_2$ , to 1 N  $H_2SO_4$  and then calcining in air at 575—650 °C.

We have synthesized a solid superacid with an acid strength of  $H_0 < -16 \cdot 04$ , the strongest surface-acid system known, and which can be used at temperatures of up to 650 °C; this catalyst also showed a superacid strength of  $H_0 \wedge -13 \cdot 75$ , with heat-treatment, even at 800 °C.

The catalyst was prepared as follows.  $Zr(OH)_4$  was obtained by hydrolysing  $ZrOCl_2 \cdot 8H_2O$  and  $ZrO(NO_3)_2 \cdot 2H_2O$  with aqueous ammonia, washing, drying at 100 °C, and powdering the precipitates below a 100 mesh. The commercial hydroxide  $Zr(OH)_4 \cdot nH_2O$  (Nakarai Chemicals, Ltd.) was also used. The hydroxides (2 g) were exposed to 1N  $H_2SO_4$  (30 ml) on a filter paper followed by drying, calcining in a Pyrex tube in air for 3 h, and finally sealing in an ampoule until use. The catalysts thus prepared from  $ZrOCl_2 \cdot 8H_2O$ ,  $ZrO(NO_3)_2 \cdot 2H_2O$ , and the commercial  $Zr(OH)_4 \cdot nH_2O$  are referred to as ZrO2-I, -II, and -III, respectively.

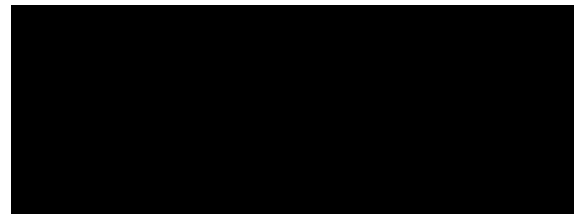
FIGURE. Reaction of butane over  $ZrO_2$ -I (O),  $ZrO_2$ -II (0), and  $ZrO_2$ -III ( $\wedge$ ): solid lines, at 180 °C; dashed line, at 130 °C. Propane yields A (17%), B(10%), C(4%), D(0%).

The catalytic activities for the reaction of butane ( $C_4$ ) were examined and the results are shown as a function of calcination

và butane) bằng cách cho  $Zr(OH)_4$  (chất này được điều chế bằng phương pháp thủy phân  $ZrOCl_2$  và  $ZrO(NO_3)_2$ ), tiếp xúc với 1 N  $H_2SO_4$  và sau đó nung trong không khí ở 575—650 °C.

Chúng tôi đã tổng hợp siêu axit rắn với độ mạnh....., hệ axit bề mặt mạnh nhất hiện nay, và có thể dùng được ở nhiệt độ lên đến 650 °C; chất xúc tác này cũng có độ mạnh là....., khi xử lý nhiệt, ngay cả ở 800 °C.

Quá trình điều chế chất xúc tác như sau. Trước hết chúng tôi điều chế  $Zr(OH)_4$  bằng cách thủy phân  $ZrOCl_2 \cdot 8H_2O$  và  $ZrO(NO_3)_2 \cdot 2H_2O$  bằng dung dịch amoniac, rửa, sấy khô ở 100 °C, và nghiền bột các chất kết tủa bằng lưới 100 mesh. Chúng tôi cũng sử dụng hydroxit thương mại  $Zr(OH)_4 \cdot nH_2O$  (Nakarai Chemicals, Ltd.) Các hydroxit được cho tiếp xúc với 1N  $H_2SO_4$  (30 ml



temperature of the catalyst in the Figure. The reaction was carried out in a microcatalytic pulse reactor with a fixed-bed catalyst (flow rate of He carrier gas 10 ml min<sup>-1</sup>; pulse size 0-05 ml; catalyst 0-3 g). The catalyst was again heated at 400 °C for 1-5 h in the He flow before reaction. Effluent products were directly introduced into a gas chromatographic column for analysis (Porapak R-2m, at 110 °C). Conversions were taken as the average from the first to the fifth pulse value. Since conversions increased until the fifth pulse for the reaction over ZrOa-I at 130 °C, the average values from the sixth to the tenth pulse reaction are shown. The maximum activity was observed with calcination at 625—650 °C for ZrO<sub>2</sub>-I, 575 °C for ZrO<sub>2</sub>-II, and 650 °C for ZrO<sub>2</sub>-III. The products were isobutane (i-C<sub>4</sub>) and propane (C<sub>3</sub>). Propane was observed as a minor material when conversions were >20% at 180 °C. Only isobutane was formed over ZrOa-I at 130 °C.

The reaction of butane was carried out in a recirculation reactor at 25 °C over ZrO<sub>2</sub>-I calcined at 650 °C, together with the catalyst treated at 500 °C for comparison [volume 170 ml; catalyst 0-3 g; butane 15 ml (normal temperature and pressure)]. The results are shown in Table 1. Pentane (C<sub>5</sub>) and isopentane (i-C<sub>5</sub>) were observed as products in addition to C<sub>3</sub> and i-C<sub>4</sub>. The amount of butane produced after 48 h, 34-2%, is close to that of the equilibrium mixture of C<sub>4</sub> and i-C<sub>4</sub> at 25 °C, 27 and 73%, respectively.<sup>2</sup>

TABLE 1. Reaction of butane over ZrO<sub>2</sub>-I at 25 °C.

a C3, C4, i-C4, C5, and i-C8 indicate propane, butane, isobutane, pentane, and isopentane, respectively. b The catalyst was heated again in air at 500 °C for 1-5 h before reaction. c The catalyst was evacuated at 250 °C for 3 h at 10<sup>-2</sup>—10<sup>-3</sup> mmHg before the reaction. d T = Trace.

The catalyst ZrO-I, heat-treated at 650 °C, also converted propane into methane and ethane at 280 °C under pulse reaction conditions (carrier of He 10 ml min<sup>-1</sup>; pulse size 0.05 ml; catalyst 0.6 g); yields of methane and ethane were 5.1 and 1.3% in the first pulse, 3.9 and 0.8% in the fifth pulse, and 3.7 and 0.8% in the tenth pulse reaction, respectively.

The acid strength of these catalysts was examined by a colour change method using Hammett indicators; the indicator is added to a powdered sample placed in sulphuryl chloride.<sup>3</sup> The results with the indicators m-nitrochloro-benzene (pK<sub>a</sub> —13.16), 2,4-dinitrotoluene (—13.75), 2,4-dinitrofluorobenzene (—14.52), and 1,3,5-trinitrobenzene (—16.04) are shown in Table 2. The acid strengths of ZrOa-I (650 °C) and ZrOa-II (575 °C) are estimated to be H<sup>0</sup> ^ — 16.04, which is higher than H<sup>0</sup> ^ —14.52 regarded as the strongest surface-acid known;<sup>3</sup> that for ZrO<sub>2</sub>-III (650 °C) being H<sup>0</sup> ^ — 14.52. It is of considerable interest that the catalyst, even when heat-treated at quite a high temperature (800 °C), is still a superacid with an acid strength higher than H<sup>0</sup> —13.75 The solid

superacids SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> and SbF<sub>5</sub>-TiO<sub>2</sub>-SiO<sub>2</sub>, whose acid strengths are higher than that of 100% H<sub>2</sub>SO<sub>4</sub> ( $H_0 = -11.9$ ) are estimated to have strengths  $-12.70 < H_0 < -11.35$  and  $-14.52 < H_0 < -13.75$ , respectively.

X-Ray photoelectron and IR spectra showed the ZrO<sub>2</sub>-I, -II, and -III catalysts to possess the bidentate sulphate ion co-ordinated to the metal. The existence of both Bronsted and Lewis acid sites was shown by the IR spectra of pyridine adsorbed on ZrO<sub>2</sub>-I (650 °C). With regard to the crystalline structure, the sulphate-treated materials were very different from those not treated in this way. The catalysts ZrO<sub>2</sub>-I (650 °C), ZrO<sub>2</sub>-II (575 °C), and ZrO<sub>2</sub>-III (650 °C) all showed the tetragonal X-ray pattern. Specific surface areas of the catalysts were much larger than those of the oxides which had not undergone the sulphate treatment.