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Sintering of alumina and effects of rare earth metal addition

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This abstract gives details on the sintering of porous alumina materials at various temperatures. The effect of adding cerium and lanthanum as a means of lowering sintering rates are also investigated. The effect of adding the cerium and lanthanum is clearly positive, with 68% of the initial surface area retained at 1,100 °C sintering after 4h, compared to 28% for the non-stabilized material. All the materials transfer from delta to theta-phase during the high temperature treatment.

1. Scope (11-point boldface)

There is a clear need in the application of catalysts to maintain high surface areas of the support materials, also at high temperatures. This has made the investigation in stabilizing alumina quite common. The stabilization is normally performed by adding a stabilizer with high melting point during the preparation of the alumina prepared for instance by precipitation [1]. The stabilizer is added in amounts ranging from 1-15% by weight. In this abstract, another approach is described. Here the stabilizing material is added to an already finished catalyst support via incipient wetness addition. The starting material is a delta alumina (Sasol GmbH), having a specific surface area of 120 m²/g as measured using nitrogen adsorption at liquid nitrogen temperature and calculated using the BET-relation. The desorption pore volume using the BJH equation [2] was 0.72 ml/g and the average pore size was 24 nm. To prepare the samples, a volume of DI-water, corresponding to the pore volume of the sample was measured and to it, an amount of salt corresponding to 1% of Ce and 1% of La in the final preparation was added. The material was dried in a hot air oven for 12 h at 120 °C. There after the material was sintered in air at 1,100 °C for 4 hours in a muzzle furnace. In addition, the virgin material was sintered at 750, 800 and 1,000 °C for comparison. The resulting materials were analyzed using powder x-ray diffraction and the resulting alumina crystal structure verified.

2. Results and discussion

The sintering of the non-stabilized sample is quite severe as temperature is increased. There is not much change in the original material at 750 or 800 °C and the surface area only drops by 3% for the latter temperature. While it is unaffected by the first, lower temperature. However, when increasing the heat treatment temperature further, there is a quite sharp decrease in surface area. When the temperature is increased to 1,100 °C the surface area drops to 33 m²/g or a mere 28% of the initial value. At the same time, the pore volume of the sample drops from the initial 0.72 ml/g to 0.22 ml/g and the average pore size increased to 28 nm.

However, for the material stabilized by Ce and La added through incipient wetness there is a vast difference for the most part. The average pore size is actually larger than for the non-stabilised sample coming in at 29 nm. Also the values for surface area and pore volume is higher than for the non-stabilized value.

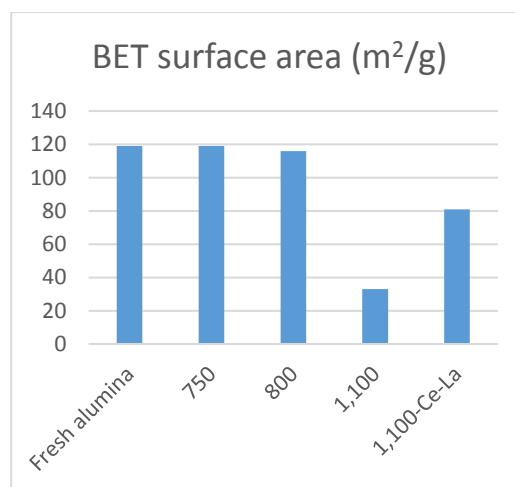


Figure 1. The results of the stabilization of the alumina support material.



The surface area is retained at 68% of the initial value or 81 m²/g. The pore volume on the other hand is retained at 0.60 ml/g. The reports of the sintering is illustrated in figure 1 with respect to surface area and summarized in table 1. All samples sintered at 1,100 °C show a theta-phase crystal structure.

Table 1 Surface area, pore volume and pore diameter of samples with varying sintering temperature.

Sample	BET surface area (m ² /g)	BJH desorption pore volume (ml/g)	Pore diameter (4V/A BET) nm	Crystal form of alumina
Fresh alumina	119	0.72	24	Delta
750	119	0.73	25	Delta
800	116	0.73	25	Delta
1,100	33	0.22	28	Theta
1,100 La Ce	81	0.60	29	Theta

3. Conclusions

The experimental work performed clearly show that a pre-prepared alumina can be stabilized using incipient wetness addition of Ce and La. Adding the stabilizing material in low amounts, 1% of each Ce and La in the resulting catalyst carrier, makes it much more resilient to sintering. This implies that the post addition of the alkaline earth metals stabilizes the material, despite the transition from one phase to another; it is thus not a stabilization due to increasing the phase-transition temperature but a true stabilization also after the phase transition.

References

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