90-098891/13 H04 ESSO 18.12.87
*US 4900-707-A
EXXON RES & ENG CO
13.12.88-US-283658 (+US-134698) (13.02.90) B01j-27/12

**Wax isomerisation catalyst mfr. - by depositing metal on support, fluorinating, crushing, sizing and activating**

**C90-043471**

Catalyst for use in a wax isomerisation process is produced by (a) depositing a hydrogenation metal component (I) on a refractory metal oxide particle or extrudate support (II), followed by calcination;

(b) fluorinating the metal-loaded support to a level of 2-10 (2-8) wt.% F, using an acidic fluorine source (III);

(c) crushing to expose the inner surfaces of the prod. and sizing to a particle size 1/32 in. or less across the largest continuous cross sectional dimension; and

(d) activating by heating in a hydrogen atmos.

In a process variant, step (c) precedes step (b).

USE/ADVANTAGE

The catalyst gives an improved yield of isomerised prods., and lube oil fractions taken from the isomerate have exceptionally high viscosity. The wax feed pref. contains 5-15%, esp. 7-10%, oil.

H(4-A7, 4-A10, 4-E, 4-E8, 4-F2A, 4-F2E) N(2-F2, 4-D, 6-E)

PREFERRED METHOD

(I) is Pt, deposited by a conventional method on (II), which is esp. alumina (gamma or eta); calcination takes place at 350-500 deg. C. Metal loading is 0.2-0.6 wt.%. (III) is aq. HF introduced e.g. by soaking or spraying, followed by drying and crushing; sizing is to within the range 1/64-1/32 in. Activation takes place at 350-500 deg. C in 1-48 hr.

EXAMPLE

1/16 in. commerical 0.6 wt.% Pt on gamma alumina extrudate was fluorided using 11.6 wt.% aq. HF to a fluoride level 8.3 wt.%, dried at 150 deg.C, crushed and sized to a 14/35 mesh (ca. 1/30 in.). It was then activated in flowing hydrogen by heating from ambient to 100 deg. C in 2 hr, to 450 deg. C in 3 hr., and holding at 450 deg. C for 1 hr.

A hydrotreated slack wax was isomerised over this catalyst at 320-347 deg. C; it gave unexpectedly better performance than did a catalyst obtained with omission of the crushing and sizing.(6pp1644CGDwgNo0/0).

89-180132/25 H04 ESSO 18.12.87
EXXON RES & ENG CO *EP -321-299-A
18.12.87-US-134698 (21.06.89) B01j-23/40 B01j-37/26 C10g-45/62

**Prodn. of catalyst for isomerising wax - comprising catalyst on metal oxide support and hydrogenation metal, by calcining fluoriding, crushing and activating**

C89-079540 R(BE DE ES FR GB IT NL)

A catalyst for use in wax isomerisation is prepd. by

(1) depositing a hydrogenation metal component on a refractory metal oxide particle or extrudate support, followed by calcination;

(2) fluoriding the loaded support, using an acidic F source;

(3) sizing the halogenated, loaded support to expose inner surfaces and give sized particles not more than 0.79375 mm across the longest continuous cross-sectional dimension; and

(4) activating by heating in $H_2$.

Opt. stage (3) may precede stage (2).

ADVANTAGE

High selective conversion of wax to liq. isomerised prod. is achieved, and the isomerised dewaxed oil prod. has high VI. Prods. obtd. from wax contg. 7% of oil have higher VI than do prods. from wax with 0% oil or 20% oil.

H(4-A7, 4-E, 4-F2A, 4-F2E) N(1-C2, 2, 4-D, 6-E)

PREFERRED CATALYST

The metal is 0.1-5 (0.2-0.6) wt. % of a Gp. VIII metal, e.g. Pt. The support is, or contains, $Al_2O_3$. Fluoridising is with a soln. of HF, to give 2-10% of F. The support is sieved to remove particles above 0.79375 mm; the particle size is pref. 0.3969-0.79375 mm. Activation is at 350-500 deg. C in $H_2$ for at least 1-48 h.

EXAMPLE

(A) A 1/16 inch $\gamma$-$Al_2O_3$ extrudate carrying 0.6 wt. % Pt was fluorided in 11.6% aq. HF soln., washed, dried at 150 deg. C in a vacuum, crushed to particles of 1/30 inch, and activated in 50 psi flowing $H_2$ by heating from room temp. to 100 deg. C in 2 h, to 450 deg. C in 3 h, and at 450 deg. C for 1 h.

(B) The catalyst used was to isomerise 600N slack waxes cong. (i) less than 1%, (ii) 7%, or (iii) 23% of oil. Conversion to 370+ deg. C prods. was: (i) 13, (ii) 24, (iii) 12.8; 25.8. The viscosity at 100 deg. C was: (i), (ii), (iii) 4.8; and viscosity index was: (i) 148, (ii) 150, (iii) 135, 137 .

(9pp510CGDwgNo0/0).

(E) ISR: No Search Report.