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A novel method of Al-pillared montmorillonite preparation for potential industrial up-scaling

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Abstract

A novel method of preparing Al-pillared montmorillonite has been developed. The process uses a minimum amount of liquid and is simpler to operate than the conventional method of pillaring. Here, the pillaring agent (solid Al_{13} nitrate) is ground together with the powdered raw montmorillonite (from Wyoming) and dialysed against a limited volume of deionised water. The resultant Al-intercalated clay is heated at 300 °C and characterized by X-ray diffraction, nitrogen sorption, and chemical analysis. The results are compared with those obtained for Al-pillared inter-layered clays (PILC) prepared by conventional procedure. Besides yielding a highly microporous and crystalline products, the new method offers the potential for extension to an industrial-scale process.

Keywords: Al-pillared clays; Industrial pillaring process; Al_{13} nitrate solid; Chlorhydrol; Dialysis

1. Introduction

Pillared inter-layered clays (PILC) have attracted increasing attention, particularly from industry since the 1970s, because of their microporous nature and catalytic potential. However, PILC have not been used as commercial catalysts. This is because the pillaring process, developed in the laboratory, is difficult to extend to an industrial scale. The laboratory method is essentially based on mixing a dilute clay suspension with a dilute pillaring solution. This laborious and time-consuming procedure involves the following steps:

(1) dispersing the clay (1% to 2%) in water;
(2) preparing a dilute pillaring solution;
(3) slowly adding the pillaring solution to the clay suspension;
(4) washing by repeated dispersions–centrifugations;
(5) final washing by dialysis followed by a last centrifugation;
(6) drying the centrifuged concentrated paste;
(7) finally, heating the dried product to produce the pillared clay.

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Table 1
Summary of experiment conditions of literature on concentrated pillaring

Reference	1	2	3	4	5	6	7	8	9	10	11	12	13
Clay	Mt	Mt	Ht, Sap	Ht, Sap Lap	Sap	Mt	Mt Ilite- Sm	Mt	Mt	Mt Sap	Mt, Lap Sap, Bd	Mt	Sap
OH/Al	1.6	2	2.4	1 and 2	1 and 2	2	2	Not precise	2	1.9	Not precise	Not precise	Not precise
$[Al]_f$ (M)	0.1	0.1	0.07	0.8	0.5	Not precise	Al-Cu	Chlorhyd.S 50%	Chlorhyd.S 50% 2.5 M	0.068 or chlorhyd.S	Chlorhyd.P	Locron (Al-hydr.chlor.) 50%	Not precise
Clay/ water	40% in DB	10% in DB	1% in DB	Different Cc	P	P and 10%	P and 33% in DB	50% in water in acetone	10%, 15%, 20%, 40%, 50%	P or suspension	50% in acetone	50% in acetone	P in DB
Al meq/g clay	25, 50, 75, 100	20, 40, 60, 70	Not precise	Not precise	23	9	30, 60	30	15	11.4	30	5, 10, 30	Not precise
Time of exchange	24 h, 48 h	1, 3, 7 days	48 h	24 h	1 night under reflux	24 h	48 h	17	7 min in microwave	12 h	24 h	2 h	0 h
W/D	2 D	1 D	D until Cl^- free	W until Cl^- free	5 W	4 W	5 D	4 W	W until Cl^- free	Filter-press	4 W	D until Cl^- free	5 D

(1) Molina et al. (1992); (2) Del Riego (1994); (3) Schoonheydt et al. (1994); (4) Schoonheydt et al. (1993); (5) Schoonheydt and Leeman (1992); (6) Sanchez and Montes (1998); (7) Frini et al. (1997); (8) Storaro et al. (1996); (9) Fetter et al. (1997); (10) Moreno et al. (1997); (11) Storaro et al. (1998); (12) Salerno and Mendioroz (2002); (13) Vicente and Lambert (2003).

Bd: Beidellite; Ht: Hectorite; Il: Illite; Lap: Laponite; Mt: Montomrillonite; Sap: Saponite; Sm: Smectite.

$[Al]_f$: final Al concentration; Cc: concentrations; Chlorhyd: chlorhydrol; D: dialysis; DB: dialysis bags; P: powder; W: washing; S: solution.