Al/Fe-PILC catalyst: Bench-scale preparation and shaping as extrudates

Conference Paper · August 2015

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Al/Fe-PILC CATALYST: BENCH-SCALE PREPARATION AND SHAPING AS EXTRUDATES

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Introduction

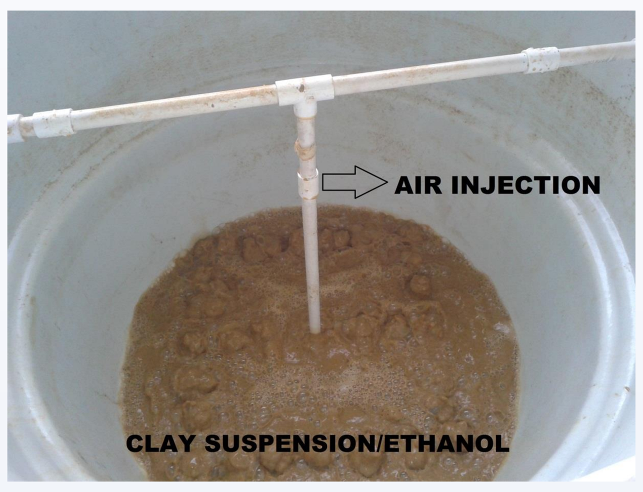
The preparation of Al/Fe-pillared clays (Al/Fe-PILC) has been extensively studied along the past few years because their low-cost, microporous structure and high catalytic performance in environmental applications *e.g.* for fixing by adsorption of different kinds of wastes, preparation of pharmaceuticals, catalytic removal of organic contaminants in aqueous effluents, formulation of coatings, among others[1]. However, scaling-up of preparation and shaping (*e.g.* as extrudates, pellets, monoliths, etc) without significant loss of catalytic properties remains being rather a challenging issue[2].

Experimental

Starting clay: Calcium-rich Bentonite (BVC) from Valle del Cauca – Colombia.

Synthesis of the Al/Fe-pillaring precursor (Al/Fe-Int)

Preparation was performed at 3.8 Kg scale by means of a procedure adapted from the already described elsewhere[1]:



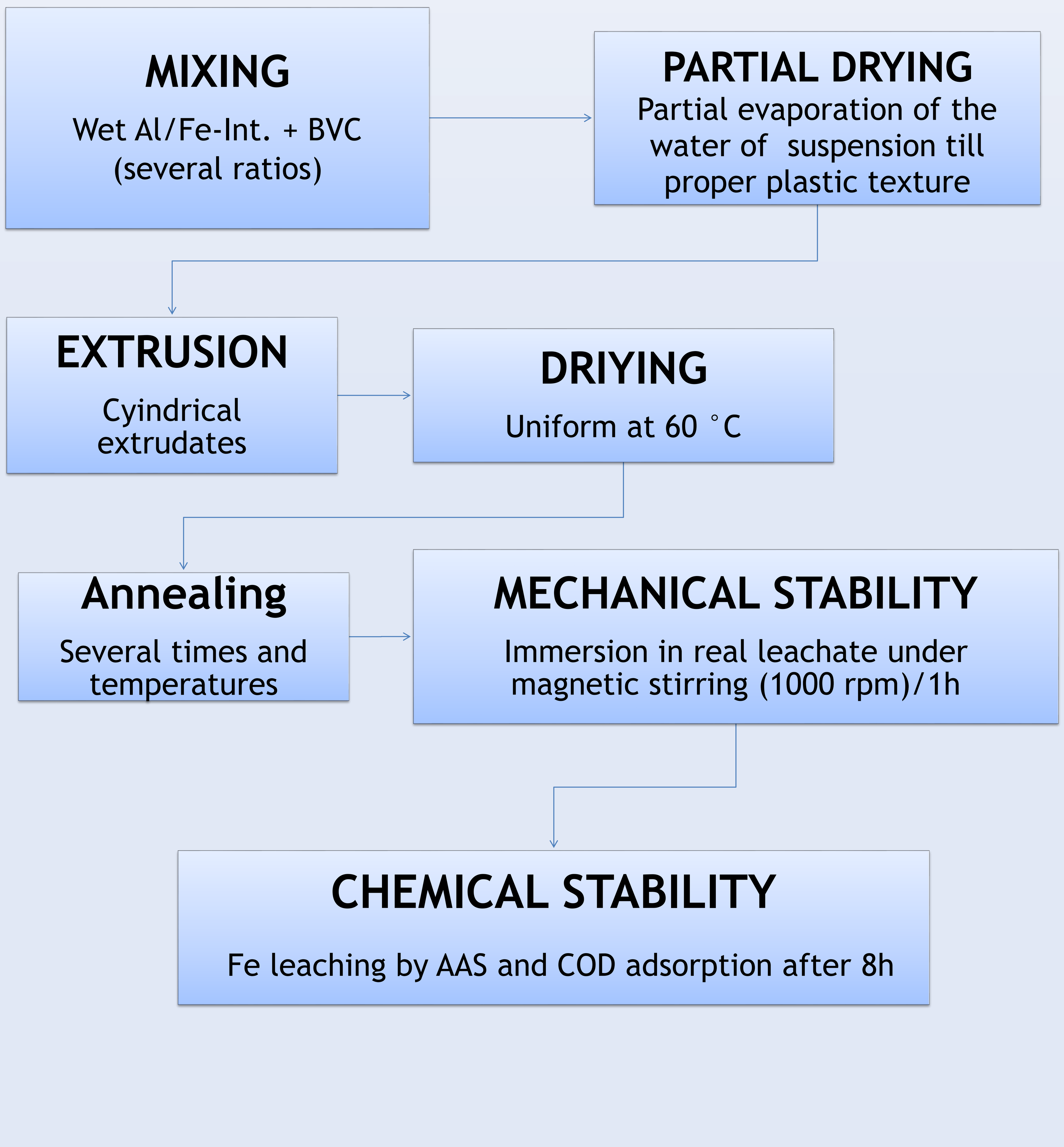
- Concentrated suspension: 50 % w/v of clay in ethanol (technical grade).
- Atomic Metal Ratio (Fe) (Fe/Al+Fe)*100 = 5.0 %
- Total Concentration of Metals [Al+Fe] = 0.6 mol/L
- Hydrolysis Ratio (OH^-/Al+Fe) = 1.6

Figure.1 Preparation of the Al/Fe-pillaring precursor

Characterization

(i) Elemental analysis (XRF) ; *(ii)* Cationic exchange capacity (CEC) – $(NH_4)^+$-exchange and *(iii)* X-ray diffraction

Preparation and analysis of extrudates



Results

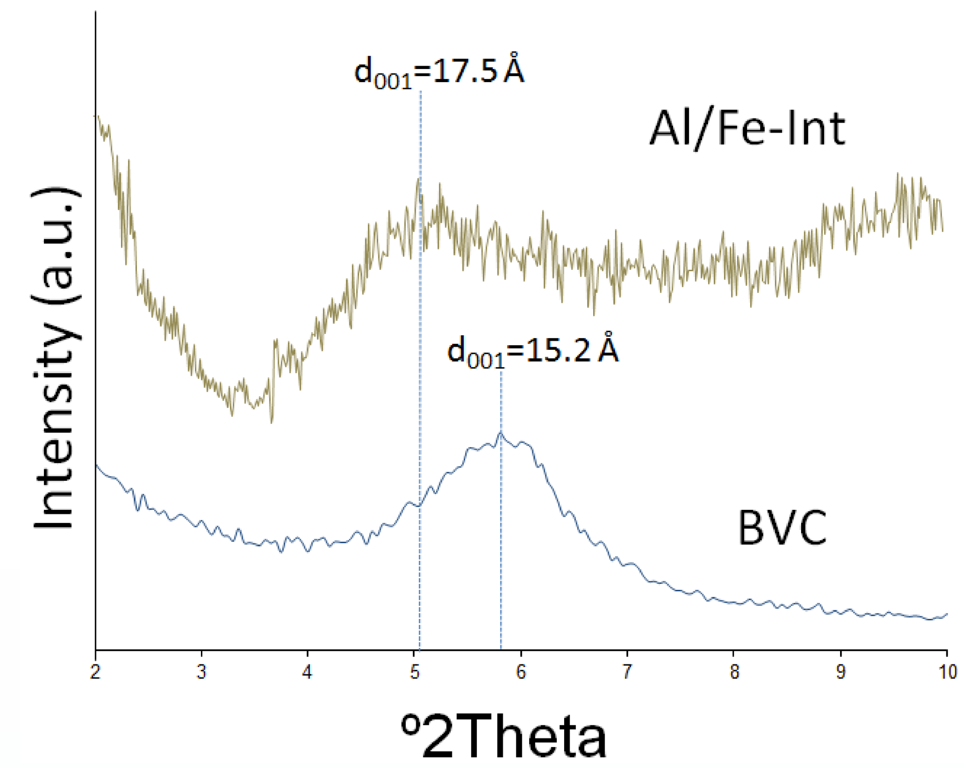


Figure.3 Powder small-angle XRD of raw bentonite (BVC) and interlayered bentonite (Al/Fe-Int).

Table 1. Main physicochemical properties of starting and Al/Fe - pillared clay*(a)*

Sample	SiO_2 (% w/w)	Al_2O_3 (% w/w)	Fe_2O_3 (% w/w)	CEC (meq./100g clay)
BVC	46.0	20.3	8.50	64
Al/Fe-BVC*(b)*	49.7	26.8	11.2	38

(a) Dry basis (110 °C); (b) Calcined at 500 °C/2 h.

Table 2. Mechanical and chemical stability of extruded materials after testing

Composition ratio AlFe-Int:BVC:water +	Thermal Treatment	Mechanical Testing(a)	Chemical Stability	
	T/t (°C/h)	Particle sizes over sieve 16 (w/w %)(b)	[Fe] (ppm)	COD adsorbed (mg O_2/g extr.)
31:31:38	500/2	97	0.5	24
31:31:38	400/2	17	ND	----------
43:19:38	**500/2**	**96**	**0,3**	**21**
43:19:38	**400/2**	**97**	**ND**	**41**
50:12:38	500/2	94	1.0	17
50:12:38	400/2	-----------	ND	----------
56:6:38	500/2	90	0.7	0
56:6:38	400/2	-----------	ND	----------
59:3:38	500/2	84	ND	0
59:3:38	500/4	82	ND	35
61:1:38	500/2	87	ND	11
61:1:38	500/4	85	ND	0
62:0:38	500/2	86	0.7	18
62:0:38	400/2	----------	1.0	----------

(a) Immersion in leachate 1h/1000 rpm: (b) Sieve ASTM 16 = (ϕ > 1.18 mm); ND = Not detected (AAS).

Conclusion

The pillaring was successful at 3.8 Kg scale (see Fig. 3 and Table 1). Heating at 500 °C produced the best mechanical properties in the extrudates irrespective the ratio Al/Fe-Int:BVC (active phase:binder); besides, these extrudates exhibited higher capacity of adsorption when the fraction of the binder (raw clay) was increased. A mass ratio 43/19/38 for active phase/binder/water was found to be the optimal in terms of both, mechanical and chemical stability (against Fe leaching) of the extrudates.

Figure 2 Final extrudates

Acknowledgement

Financial Support by VIPRI-Universidad de Nariño (Project code 558/2012) is highly appreciated.

References

[1] Galeano L.A., Vicente M.A., Gil A., *Catal. Rev. Sci & Eng.* 56 (**2014**) 239– 287.

[2] Mohino F., Martin A.B., Salerno P., Bahamonde A., Mendioroz S. *Appl. Clay Sci.* 29(2) (2005) 125-136.