CHAPTER-3

RESULTS AND DISCUSSION

3. RESULTS AND DISCUSSION

3.1. Preparation and Characterization of Iron (III) Silicates

As mentioned in the experimental part, the Na^+-forms of iron (III) silicates as a cation exchange materials were prepared by adding equimolar solution of iron trichloride to sodium silicate to obtain different Fe/Si molar ratios of 1.0, 0.5 and 2.0 for FeSi-I, FeSi-II and FeSi-III, respectively, as in Table (6). All samples of iron silicates (FeSi-I, FeSi-II and FeSi- III) were hard granulates in nature and suitable to use in packed column. Both ratios of iron silicates still exhibit their original brown after the rough washing with distilled water and drying at 50 °C. Gradual change in color from brown to reddish brown to dark brown were observed when the prepared materials heated from 50±1 to 400 °C and finally dried at 850°C. No changes in particle diameters of iron silicate samples are observed with increasing the heating temperatures up to 850 °C. Physicochemical properties of the prepared materials of iron silicate (FeSi-I, FeSi-II and FeSi-III) were identified by using different techniques, such as, chemical stability, IR-spectra, X-Ray fluorescence and X-Ray diffraction patterns as well as TG and DTA thermal analysis.

3.1.1. Chemical stability

Chemical stability of synthetic inorganic ion exchangers plays an important role in their analytical applications. Exchangers which have a high solubility in water as well as in acid media, may not be useful for separation investigations[122-125]. It is therefore advisable to have enough guide of the solubility of an ion exchanger. In this concern, the chemical stability of the prepared iron silicate samples (FeSi-I, FeSi-II and FeSi-III) (Table 7) was investigated in water, nitric and hydrochloric acids at different concentrations (0.1, 0.5, 1.0, 2.0, and 3.0 M acid). The results

Table (6): Chemical formula and some physical properties of the obtained iron silicates.

Exchanger	Reactant molar ratio Fe : Si	Chemical formula	M.Wt.	XRD	Color
FeSi-I	1 : 1	$(Fe_2O_3)_{1.14}.SiO_2.\ 3.8H_2O$	264.45	Amorphous	D.B
FeSi-II	1 : 2	$(Fe_2O_3)_{0.55}.SiO_2.1.3H_2O$	172.22	Amorphous	D.B
FeSi-III	2 : 1	$(Fe_2O_3)_{1.83}.SiO_2.\ 6.0SH_2O$	460.90	Amorphous	D.B

D.B*= dark brown

Table (7): Chemical stability of different iron (III) silicates (g/l) at 25±1°C.

Exchanger	Solubility, mg/L											
	H_2O	HNO_3, M						HCl, M					
		0.01	0.1	0.5	2.0	3.0	4.0	0.01	0.1	0.5	2.0	3.0	4.0
FeSi-I	B.D	0.011	0.023	0.034	0.046	P.D	C.D	0.028	0.038	0.049	0.066	P.D	C.D
FeSi-II	B.D	0.007	0.013	0.021	0.035	0.091	C.D	0.012	0.024	0.042	0.059	P.D	C.D
FeSi-III	B.D	0.017	0.029	0.040	0.61	P.D	C.D	0.027	0.047	0.059	0.087	P.D	C.D

C.D: Complete dissolved B.D: Below detection P.D: Partially dissolved