RESULTS AND DISCUSSION

3. RESULTS AND DISCUSSION

Inorganic ion exchangers of high selectivity can be used as adsorbents and additives in solidification of liquid wastes. The increasing concern towards protection of the environmental and optimization of a wide range of industrial in both nuclear and non-nuclear fields imposes the need to find and synthesis new highly selective ion-exchangers materials. Among these, several new synthetic ion exchange materials, double salts of polybasic acids with tetravalent metal type such as silico(IV) titanate (SiTi) is the promising material for use in removal of toxic elements from liquid waste solutions. This study aims to a better understanding of the various phenomena underlying these behaviors.

Characterization of this material using X-ray diffraction patterns and infrared spectroscopy was conducted. Capacity, equilibrium measurements, selectivity patterns for Cs^+, Na^+, Co^{2+} and Sr^{2+}, were determined at different conditions on this material. Such investigations are essential to assess the use of the prepared inorganic sorbent in various fields.

3.1. Preparation of silico(IV) titanate:

As a result of the method of preparation, the granular types of silico(IV) titanate cation exchanger was prepared by dropwise addition of equimolar solutions of sodium metasilicate (0.6 M) to titanium tetrachloride (0.6 M) at Si / Ti molar ratio of 1.0 with continuous stirring in water bath at 60±1 °C. The mixed solutions were immediately hydrolyzed in deionized water at 60±1 °C. The white precipitate of silicotitanate was formed by addition of few drops of ammonia solution. Then the precipitate was left to age in the mother liquor overnight, filtered and then washed with deionized water. The white precipitate was dried at 60 °C for 4 days and rewashed with near boiling deionized water (70±1 °C) in order to remove the air trapped and adherent fine particles, the white product was ground and sieved to obtain a mesh size of 0.212 – 1.00 mm and stored at room temperature. No change in particle sizes of SiTi due to thermal treatment was observed.

3.2. Chemical Stability

The weight losses for the prepared silicotitanate at different drying temperatures from 200 to 850 °C are shown in **Table (3)**. These weight losses correspond to the water content removal of the exchanger at these temperatures.

The chemical stability of the exchanger towards water and acidic medium is one of the most important factors which controlling the possibility of application of the prepared ion exchanger in the separation technology purposes. So, the chemical stability of SiTi was studied in water and different acid concentrations of HNO_3 and HCl [0.1, 1, 2, 3, 4, 5 and 6 M]. The results of solubility of silicotitanate in water and different concentrations of HNO_3 and HCl are given in **Table (4)**. From this Table, it was found that the silicotitanate has high stability in water and HNO_3 solution up to 6 M. While the exchanger was physically quite stable up to 4 M HCl, after which it completely dissolved at 6M HCl. It is

TABLE (3): Weight losses of the prepared silicotitanate as a function of temperatures.

Ion Exchanger	Heating Temperatures, (°C)	Water Losses, (w/w %)
SiTi	200	9.64
	400	13.92
	650	19.75
	850	23.96

TABLE (4): Solubility of the prepared ion exchanger (SiTi) in defferent media (g / l) at 25±1 °C .

Medium	Solubility (g / L)													
			0.1M		1M		2M		3M		4M		5M		6M	
	Si	Ti	Si	Ti	Si	Ti	Si	Ti	Si	Ti	Si	Ti	Si	Ti	Si	Ti
H_2O	UD	UD	-	-	-	-	-	-	-	-	-	-	-	-	-	-
HNO_3	-	-	UD	UD	0.003	0.002	0.003	0.011	0.006	0.02	0.009	0.029	0.02	0.05	0.03	0.07
HCl	-	-	0.003	0.005	0.006	0.004	0.009	0.02	0.015	0.16	0.11	0.21	S	S	D	D

UD : Under Detection
S : Partially Soluble
D : Completely Soluble

clear that the solubility values were reproducible within ± 10 % for Si and Ti from its original values.

3.3. Characterization of the prepared SiTi

The prepared silicotitanate (SiTi) was characterized using different analytical techniques such as XRD, FTIR, DTA – TG analysis and surface area measurements.

3.3.1 Infrared (IR) spectra

The skeletal structure of the prepared silicotitanate dried at different temperatures (50, 200 and 400 ^{o}C) was defined using infrared spectrophotometer technique as shown in **Fig. (1)**. It was found that, the infrared spectra of silicotitanate sample showed a broad peak at ~3400 cm^{-1}. This broad peak is characteristic to the stretching mode of free water and OH groups adsorbed on the titanate sample (133). The strong peak observed at ~ 1630 cm^{-1} represents the bending mode of water molecules adsorbed on SiTi (114, 115). The last two peaks at ~ 1410 cm^{-1} and ~ 500 cm^{-1} are due to the Ti – OH deformation vibration and Ti – O bonds in the structure, respectively (116). It was found that, the band intensities of molecular water at about 3400 and 1630 cm^{-1} was decreasing by decreasing water content of the ion exchanger parallel to the increase in heating in temperature from 50 to 400 ^{o}C as shown in **Fig.(1)**.