



Cadmium leaching from thermal treated and gamma irradiated Mexican aluminosilicates

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ABSTRACT

Thermal and radiation effects on the leaching of cadmium from two cadmium exchanged zeolitic tuffs and one clay were determined. The cadmium exchanged aluminosilicates were heated at different temperatures (500, 700, 900 and 1100 °C), and the materials were then treated with NaCl (1 M and 5 M) and HNO₃ (0.001 M and 1 M) solutions to determine the leaching behaviour of cadmium from the materials. The stability of cadmium in the materials increased as the heating temperature was increased. Cadmium leaching from gamma irradiated and heated materials at 1100 °C was higher than leaching from non-irradiated samples.

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1. Introduction

Cadmium is used in reactor components such as control rods and shields to adsorb neutrons, resulting in the formation of various radioactive isotopes by (n,γ) neutron activation reactions, other radioactive isotopes of cadmium are produced as fission products [1]. These radioactive isotopes of cadmium also are present in spent nuclear fuel and radioactive wastes associated with the operation of nuclear reactors and fuel reprocessing plants. The reprocessing wastes solutions from LWR fuel irradiated to a fairly typical burn up contain 115 mg/kg U of radioactive isotopes of cadmium [2]. ¹⁰⁹Cd, and ^{113m}Cd have half lives long enough to warrant potential concern, and their presence in environment is a potential health hazard, because of their (¹⁰⁹Cd and ^{113m}Cd) high specific activity, radioactive decay and chemical toxicity [3].

Aluminosilicates like zeolites and clays are of considerable interest in the nuclear industry, because are natural, inexpensive, efficient and selective inorganic cation-exchangers and are used as waste processing media, waste forms and potential back-fill materials in nuclear waste repositories [4].

Their ion exchange capacity is the basis for those applications, because they can remove radioactive metal ions from liquid waste. The immobilization of the exchanged material takes place when it is incorporated into cement matrices and finally in the geosphere the ion exchange retard the migration of radionuclides [5].

Because the ion exchange reaction is a reversible process, the radiation induces structural changes that may also effect the re-

lease of adsorbed radionuclides when the zeolite and clay phases are in contact with aqueous solutions [4].

However, under different conditions, the initially safe cement matrix could, indeed, present micropores and the radioactive ions could diffuse out the matrix [6–9].

One of the main problems is that these cations can be exchanged again if water is in contact with the exchanged materials. An alternative way to safely store radionuclides after entrapping them in an aluminosilicate framework is based on a thermal treatment that destroys the aluminosilicate structure and blocks the radioactive species into a vitreous lattice or a non-exchanging crystalline phase [10,11]. As these materials are intended to retain radionuclides, they will receive considerable heat decay and radiation doses over time, and the cumulative doses can be substantial.

Some studies in cation exchanged synthetic zeolites have reported that these materials are susceptible to both radiation and thermally-induced amorphization [4,5,12–14]. The same effect was observed in various types of natural cation exchanged zeolitic materials [15–17].

A consequent from the amorphization in both kind of material is enhancing the immobilization for the trapped cations in the vitrified lattice diminishing its release when the amorphized phases are in contact with aqueous solutions [5,18].

In a previous paper we reported the immobilization of cobalt in thermal treated (1100 °C) and gamma irradiated aluminosilicates samples, this effect was not observed in the unheated gamma irradiated aluminosilicates [19].

Studies related to radiation and thermal induced chemical and structural transformations in clay minerals are limited. Gu et al. [20] studied the radiation stability of smectites (montmorillonite, nontronite, and saponite) as a function of temperature, and found

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that all three phases underwent a crystalline-to-amorphous transformation when exposed to electron beam irradiation over the temperature range of 25–750 °C. A weak loss of crystallinity of kaolinite was observed by Plotze et al. [21], when irradiated various clay minerals with a ^{60}Co gamma source at a dose of 1.1 MGy. Corbett et al. [22] observed atomic displacements produced in a kaolinite structure with doses of 0.667 MeV gamma radiation, which affects the crystallinity, specific surface area and cation exchange capacity of the clay.

However, there are limited data on the effects of radiation and release of the adsorbed radionuclides, due primarily to the difficulties in studying the chemical properties of the post-irradiated materials [4].

The purpose of this paper was to determine the stability of Cd^{2+} in aluminosilicates after thermal treatment and gamma irradiation, and to compare the results with those obtained in a previous work with Co^{2+} exchanged aluminosilicates [19].

2. Experimental procedures

Two clinoptilolite rich tuffs (Zcrem and Zfran) were obtained from the Graben of Villa de Reyes in the central part of Mexico in San Luis Potosi and Guanajuato States, between the parallels 22°50' to the N and 21°07' to the S. The tuffs were in the shape of a collapsed block 200 km large by 15 km wide, filled by lacustrine and alluvial sediments and ash flow tuffs [23]. Their compositions, solid surface areas, pore volumes and average pore diameters have been reported elsewhere [24]. The kaolinite clay rock was collected from Sombrete, in the State of Zacatecas, Mexico.

The zeolitic rocks were milled and sieved with a grain size between 8 and 10 mesh. Kaolinite clay rock was crushed and sieved between 60 and 100 mesh.

2.1. Modification with sodium chloride

The materials were first treated with 5 M NaCl solution for 8 days, and washed six times with deionised water until no presence of chloride ions was indicated in the washing solution using a AgNO_3 solution. The Na-treated zeolitic rocks and Na-clay were then dried at room temperature for two days and finally the samples were left in a humid medium to equilibrate with water and the materials were labelled as Na-Zcrem, Na-Zfran and Na-kaolinite.

2.2. Cadmium exchange

Seven grams of each zeolite and clay sample initially in the sodium form were put into contact with 700 mL of a 0.5 M cadmium nitrate solution (prepared with a reagent grade, Baker hexahydrated cadmium nitrate 99%) under agitation for 24 h at room temperature. The liquid phases were separated from the solids by centrifugation and cadmium in the aqueous phases was determined by atomic absorption spectrometry [3]. The materials samples exchanged with cadmium were referred as Cd-Zcrem, Cd-Zfran and Cd-kaolinite.

2.3. Thermal treatment

One gram of each Cd-Zcrem, Cd-Zfran and Cd-kaolinite materials were then thermally treated at 500, 700, 900 and 1100 °C for 2 h.

2.4. Cadmium leaching

It was measured through lixiviation with 1 M and 5 M sodium chloride and 0.001 M and 1 M nitric acid solutions and deionised

water for 24 h. Hundred milligram of each Cd-Zcrem, Cd-Zfran and Cd-kaolinite samples, thermally treated (500, 700, 900 and 1100 °C) were shaken separately with 10 ml of each: deionised water, 1 M and 5 M sodium chloride and 0.001 M and 1 M nitric acid solutions for 24 h. The mixtures were centrifuged and the liquid phases decanted. Cadmium was analysed in the remaining solutions by atomic absorption spectrometry.

2.5. Irradiation of samples

The irradiation effects on the stability of Cd-Zcrem, Cd-Zfran and Cd-kaolinite aluminosilicates without heating and on the same materials heated at 1100 °C were determined. Samples (200 mg) were gamma irradiated with a ^{60}Co gamma source JS6500 at 767 Mrad. The gamma irradiated solids were characterized by X-ray diffraction. Irradiation effects were tested through treating the materials with different solutions. One hundred milligrams of the unheated and irradiated Cd-Zcrem, Cd-Zfran, Cd-kaolinite and 1100 °C heated irradiated samples were shaken for 24 h with 10 ml of 5 M NaCl or 1 M HNO_3 . Solids and liquids phases were separated by centrifugation and Cd^{2+} present in the NaCl or HNO_3 solutions was determined by atomic absorption spectrometry.

2.6. X-ray diffraction

Power diffractograms of the aluminosilicate samples were obtained with a Siemens D500 diffractometer coupled to a copper anode X-ray tube. Conventional diffractograms were used to identify the materials and to verify crystalline structure.

2.7. Infrared spectroscopy

IR spectra of the samples in KBr pellets were recorded in the 4000–400 cm^{-1} range using a Nicolet Magna-IR spectrometer.

2.8. Atomic absorption spectrometry

Cadmium in the aqueous phases was measured with a Perkin-Elmer 2380 atomic absorption spectrophotometer in the flame mode and the sensitivity was 0.028 mg/L.

2.9. Scanning electron microscopy

The samples were mounted directly on the holders, covered by sputtering with gold, and then observed at 10 kV and 20 kV in a XL 30 Philips electron microscope. The microanalysis was done with an EDS (energy X-ray dispersive spectroscopy) system.

2.10. Surface areas (BET)

The BET surface areas were determined by standard multipoint techniques of nitrogen adsorption, using a Micromeritics Gemini 2360 instrument. The samples were heated at 373 K for 2 h before specific surface areas were measured.

3. Results and discussion

3.1. Thermal treatment

X-ray diffraction patterns showed that Cd-exchanged Zcrem tuff is mainly composed of clinoptilolite with minor quartz content. The crystalline phase was maintained and quartz was increased in the sample treated at 500 °C. The structure of original Zcrem tuff collapsed and a quartz polymorph phase trydimite and a k-feldspar type phase like albite were found in the sample heated at 1100 °C (Fig. 1(a)–(c)).

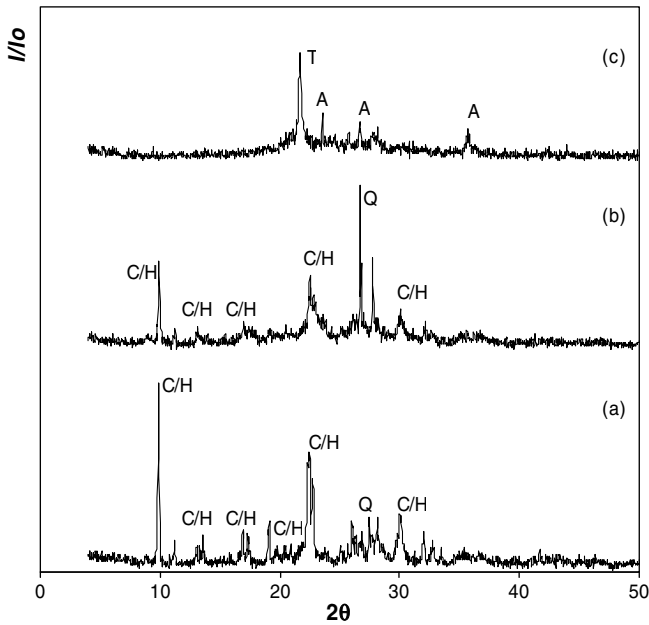


Fig. 1. X-ray diffraction patterns of Zcrem tuff: (a) Cd-exchanged sample, (b) Cd-exchanged sample heated at 500 °C, (c) Cd-exchanged sample heated at 1100 °C. C/H: Clinoptilolite-Heulandite; A: Albite; T: Trydimite; Q: Quartz.

Cd-exchanged Zfran tuff is mainly composed of clinoptilolite with a minor anorthite content. After heating the sample at 500 °C, the crystalline structure was maintained but the clinoptilolite phase diminished, and the k-feldspar albite was found. The structure collapsed and a quartz polymorph trydimite phase was the main compound found when the sample was heated at 1100 °C (Fig. 2(a)–(c)).

The Cd-exchanged clay is mainly composed of kaolinite, quartz, and wallastonite. When the sample was heated at 500 °C, kaolinite diminished but the structure did not collapse. At 1100 °C, the clay

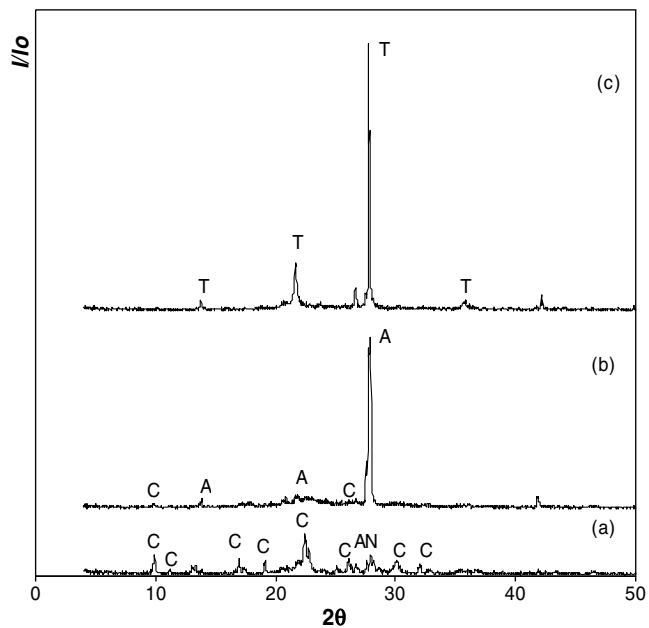


Fig. 2. X-ray diffraction patterns of Zfran tuff: (a) Cd-exchanged sample, (b) Cd-exchanged sample heated at 500 °C, (c) Cd-exchanged sample heated at 1100 °C. C: Clinoptilolite; AN: Anorthite; A: Albite; T: Tridymite.

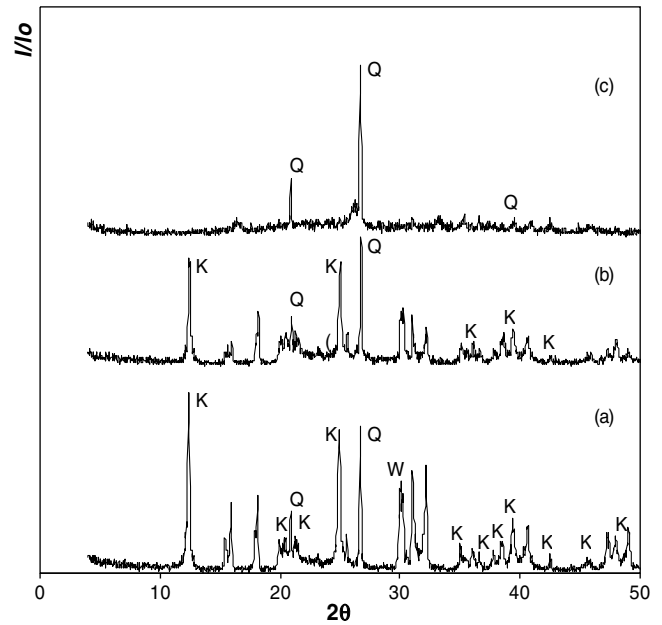


Fig. 3. X-ray diffraction patterns of kaolinite clay: (a) Cd-exchanged sample, (b) Cd-exchanged sample heated at 500 °C, (c) Cd-exchanged sample heated at 1100 °C. K: Kaolinite; W: Wallastonite; Q: Quartz.

structure collapsed and quartz was the only phase found (Fig. 3(a)–(c)).

Table 1 shows the Cd²⁺ leaching behavior from the aluminosilicates with different aqueous solutions. In general, cadmium leaching with deionized water from the zeolitic tuffs samples was not observed. For the clay samples, high leaching percentages of cadmium were observed when the samples were heated at 500 °C and 700 °C, because the clay was not destroyed at this temperature as shown in Fig. 3(b). At 1100 °C, when the clay structure collapsed,

Table 1

Cadmium leached with deionized water, 1 M and 5 M NaCl and 0.001 M and 1 M HNO₃ from cadmium exchanged aluminosilicates heated at different temperatures

Temperature (°C)	Cd ²⁺ leached (%)		
	Kaolinite	Zcrem	Zfran
<i>Deionized water</i>			
500	52.1	0.0	0.0
700	37.9	6.7	0.0
900	14.2	0.0	0.0
1100	0.0	0.0	0.0
<i>1 M NaCl</i>			
500	55.3	13.1	8.0
700	42.6	4.9	2.8
900	7.0	0.7	0.4
1100	0.0	0.0	0.0
<i>5 M NaCl</i>			
500	97.9	17.4	9.8
700	36.3	4.7	3.0
900	7.0	0.9	0.7
1100	0.0	0.0	0.0
<i>0.001 M HNO₃</i>			
500	47.4	1.6	1.5
700	33.2	1.5	1.0
900	7.8	1.0	0.7
1100	0.0	0.0	0.0
<i>1 M HNO₃</i>			
500	100.0	12.7	8.7
700	83.7	6.7	9.8
900	66.7	3.0	5.5
1100	0.0	0.0	0.0

cadmium was trapped in the amorphous solid and cadmium leaching was not observed.

The highest cadmium leaching was observed, when 1 M and 5 M NaCl solutions were put in contact with the aluminosilicates heated at 500 °C and 700 °C (Table 1), high concentration of Na⁺ may replace Cd²⁺ cations from the samples because the aluminosilicates are not destroyed at these temperatures as shown in Figs. 1(b)–3(b). High cadmium retention in samples heated at 900 and 1100 °C may be attributed to the collapse of the material structures (Figs. 1(c)–3(c)).

When the aluminosilicates were heated at 500 °C and 700 °C and treated with 0.001 M and 1 M HNO₃ solutions, a high cadmium leaching was observed which may be due to the ions exchange of H⁺ and Cd²⁺, since at these temperatures the structures are not destroyed and the Cd²⁺ ions are available for ion exchange process.

At 1100 °C the aluminosilicates were vitrified and cadmium was immobilized in the amorphous structures. The effect of high temperature is clear; the initial order is lost and the materials lose their structures.

In order to verify volatile losses of Cd⁺ by heating the cadmium exchanged materials, the content of cadmium in the samples was determined by EDS before and after thermal treatment. As shown in Table 2, losses of cadmium were not observed considering the statistical error.

After heating the cadmium exchanged materials at 1100 °C, the specific surface areas measured by BET for the zeolitic samples changed dramatically and the pore volume diminished (Table 3). These results indicate that at least partial vitrification appears to have taken place.

3.2. Cadmium leaching from gamma irradiated samples

Table 4 shows the cadmium leaching with 5 M NaCl or 1 M HNO₃ solutions, from the gamma irradiated, unheated and heated

Table 2
Weight percent of cadmium in the materials before and after heated at 1100 °C

	Unheated (25 °C)	Heated (1100 °C)
Cd-Zcrem	6.68 ± 1.8	5.49 ± 0.59
Cd-Zfran	7.80 ± 1.4	7.25 ± 0.28
Cd-Kaolinite	0.73 ± 0.12	0.86 ± 0.06

Table 3
Specific surface area and pore volume of the materials before and after heated at 1100 °C

	Specific surface area (m ² /g)	Pore volume (cm ³ /g)
Cd-Zcrem (25 °C)	17.87	0.046
Cd-Zfran (25 °C)	20.96	0.057
Cd-Kaolinite (25 °C)	9.35	0.029
Cd-Zcrem (1100 °C)	0.04	0.003
Cd-Zfran (1100 °C)	0.88	0.001
Cd-Kaolinite (1100 °C)	9.09	0.016

Table 4
Cadmium leached with 5 M NaCl and 1 M HNO₃ after thermal and irradiation treatments of cadmium exchanged aluminosilicates

Temperature (°C)	Cd ²⁺ leached (%)		
	Cd-Kaolinite	Cd-Zfran	Cd-Zcrem
<i>5 M NaCl</i>			
Unheated (25)	33.8	27.6	50.1
Heated (1100)	30.4	4.9	12.4
<i>1 M HNO₃</i>			
Unheated (25)	53.9	36.3	47.4
Heated (1100)	9.5	5.2	14.5

at 1100 °C cadmium exchanged aluminosilicates. The amount of cadmium leached with 5 M NaCl solution from the unheated gamma irradiated Zfran tuff and Zcrem tuff is about five and four orders of magnitude higher than the cadmium leached from the 1100 °C heated gamma irradiated materials respectively. Cadmium leaching was similar for unheated and heated clay samples. The cadmium leached with 1 M HNO₃ solution was about five, seven and three orders of magnitude higher for the unheated gamma irradiated kaolinite, Zfran and Zcrem tuffs than the leaching found for the 1100 °C heated gamma irradiated materials, respectively.

It has been shown that thermal treatment of aluminosilicates exchanged with different cations Co²⁺, Sr²⁺, Cs⁺ [6,12,13,15–19] at high temperatures, may result in the collapse of channel openings that can block the release of adsorbed ions.

Another possible explanation about the reduction of the release of adsorbed ions from materials thermally treated was reported by Norby et al. [25], who worked with Cs⁺ and zeolite NaY, they proposed that Cs⁺ ions in the Cs-exchanged zeolite-NaY can migrate from the supercage into the sodalite cage when the material was dehydrated at temperatures above 500 °C. Because the diameter of Cs⁺ ions (0.34 nm) is larger than the six-ring aperture (0.22 nm) in the sodalite cage, Cs⁺ would remain trapped inside the small sodalite cages.

In general, cadmium leaching was not observed in the samples heated at 1100 °C but it was observed in the heated and gamma irradiated samples. These results show that the stability of metal ions in exchanged aluminosilicates is affected by gamma radiation.

Fig. 4 shows the X-ray diffraction patterns of gamma irradiated unheated and heated cadmium exchanged kaolinite clay, there is a clear presence of potassium calcium silicate which was not observed in the non-irradiated samples (Fig. 3(a)–(c)).

The IR spectra of the unheated and heated gamma irradiated kaolinite showed that the absorption band at 916 cm⁻¹ due to OH deformation vibrations and some frequencies in the regions 400–800 cm⁻¹ and 900–1200 cm⁻¹ corresponding to Si–O stretching and mixed Si–O deformations and octahedral sheet vibration respectively [26,27] were not observed in the IR spectra of the heated gamma irradiated kaolinite clay sample (Fig. 5(a)–(b)). These results show effective damage of the materials after thermal treatment and gamma irradiation and they are in agreement with our results obtained in a previous work [19].

Fig. 6 shows that gamma irradiation of cadmium exchanged Zcrem tuff leads to the formation of the potassium calcium silicate phase in both unheated and heated samples, which was not observed for the non-irradiated samples (Fig. 1(a)–(c)).

Fig. 7 shows that k-feldspars (albite) is formed when the unheated cadmium exchanged Zfran tuff was gamma irradiated and for the heated and gamma irradiated samples, sanidine and a polymorph quartz phase (trydimite) were found.

The IR spectra of heated gamma irradiated zeolitic tuffs and unheated gamma irradiated samples (Figs. 8(a) and (b) and 9(a) and (b)) showed the disappearance of some frequencies in the region of (600–800 cm⁻¹) corresponding to a weak vibration due to symmetric stretching [28,29], which suggests a dealumination and destruction of the zeolitic tuffs [30].

Also, the absorption band of vibration frequencies due to asymmetric stretching present at (1070–1080 cm⁻¹) [28,29] in the unheated zeolitic tuffs samples, was shifted towards higher frequencies (1100 cm⁻¹) in the IR spectra of the heated gamma irradiated samples, which indicates a partial structural breakdown, accompanied by dealumination [30].

The typical bands of water observed in the unheated zeolite tuff materials corresponds to the broad band characteristic of hydrogen-bonded OH at about 3400 cm⁻¹ and the bending vibration of water at 1645 cm⁻¹ [28]. Lower relative intensities in the same band frequencies in the IR spectra of heated gamma irradiated

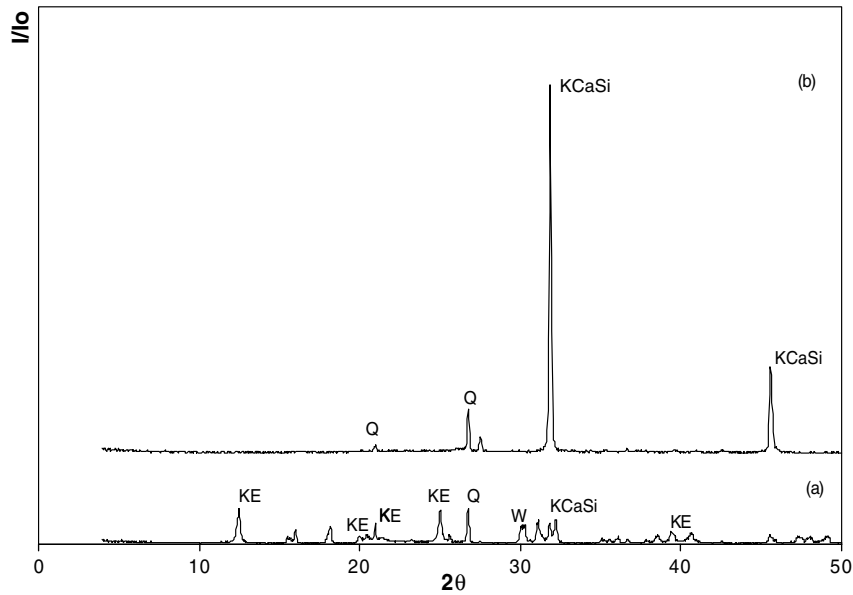


Fig. 4. X-ray diffraction patterns of gamma irradiated kaolinite clay: (a) Cd-exchanged sample unheated, (b) Cd-exchanged sample heated at 1100 °C. KE: Kaolinite; W: Wallastonite; Q: Quartz; KCaSi: Potassium calcium silicate.

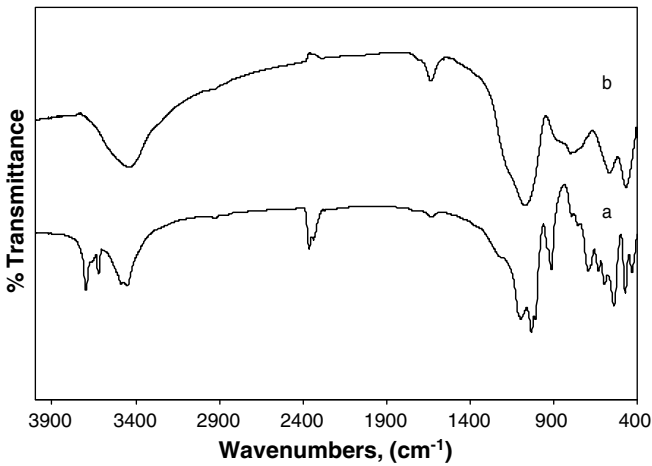


Fig. 5. IR spectra of gamma irradiated kaolinite clay samples: (a) unheated, (b) heated at 1100 °C.

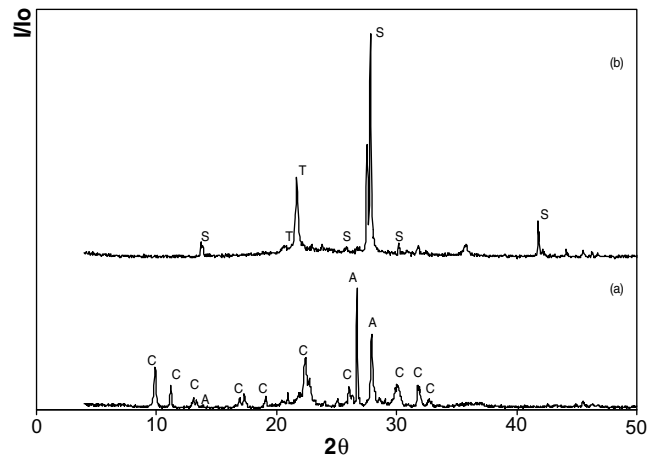


Fig. 7. X-ray diffraction patterns of gamma irradiated Zfran tuff: (a) Cd-exchanged sample unheated, (b) Cd-exchanged sample heated at 1100 °C. C: Clinoptilolite; A: Albite; S: Sanidine; T: Trydimite.

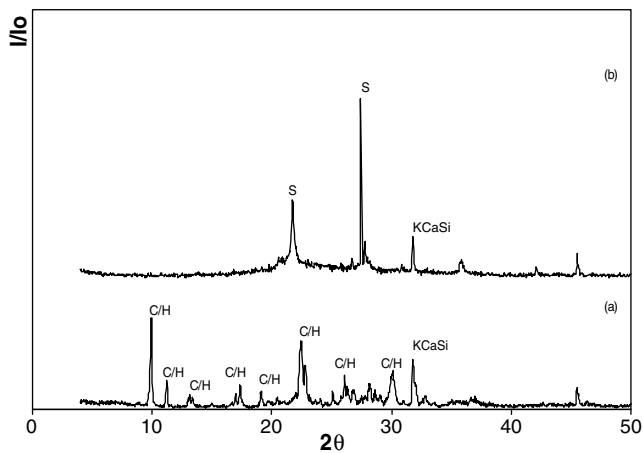


Fig. 6. X-ray diffraction patterns of gamma irradiated Zcrem tuff: (a) Cd-exchanged sample unheated, (b) Cd-exchanged sample heated at 1100 °C. C/H: Clinoptilolite-Heulandite; S: Sanidine; KCaSi: Potassium calcium silicate.

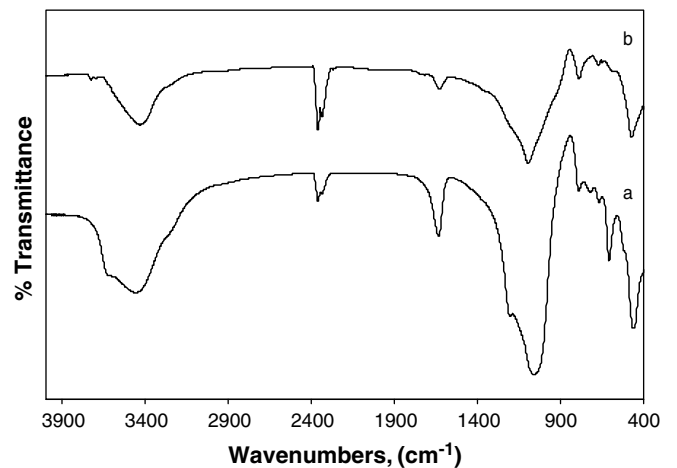


Fig. 8. IR spectra of gamma irradiated Zcrem tuff samples: (a) unheated, (b) heated at 1100 °C.

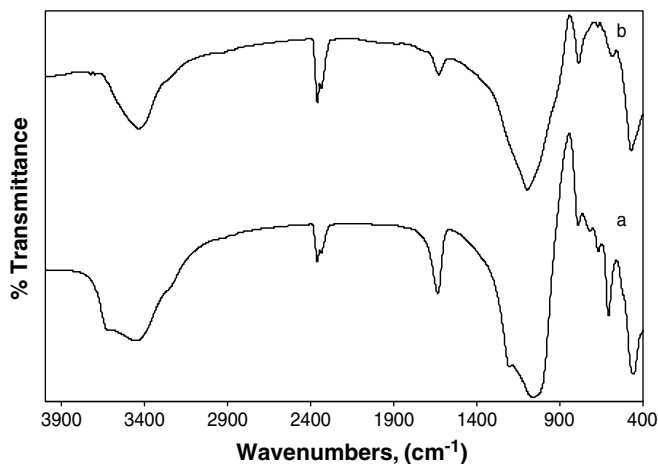


Fig. 9. IR spectra of gamma irradiated Zfran tuff samples: (a) unheated, (b) heated at 1100 °C.

materials were found. Band frequency at 2360 cm^{-1} is associated to some impurity of the KBr since this band was observed in the KBr pellets without any sample.

It has been reported [6,31] that some structures submitted to high doses of irradiation, may develop defects or recrystallize as new phases and the formation of composite materials are different from those obtained with thermal treatments.

4. Conclusions

It was found that the cadmium was best immobilized in the zeolitic tuffs and kaolinite clay when they were heated at temperatures higher than 700 and 900 °C, respectively. At 1100 °C, cadmium was trapped firmly in both kind of aluminosilicates, this is in agreement with our results in a previous research with cobalt [19].

The cadmium leaching from gamma irradiated and heated (1100 °C) aluminosilicates was higher than cadmium leaching for heated (1100 °C) non-irradiated samples, this may be due to the formation of potassium calcium silicate and other new phases due to the damage produced by gamma irradiation. These results show the importance to study the effects of the radiation in this kind of cation exchanged aluminosilicates, because the vitrification by thermal treatment may be insufficient for long term storage in a nuclear waste repository.

Acknowledgements

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