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Bitumen Coating as a Tool for Improving the Porosity and Chemical Stability of Simulated Cement-Waste Forms

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ABSTRACT

Coating process of simulated cement-based waste form with bitumen was evaluated by performing physical and chemical experimental tests. X-ray diffraction (X-RD), Fourier transform infrared spectroscopy (FT-IR) and electron microscope investigations were applied on coated and non-coated simulated waste forms. Experimental results indicated that coating process improved the applicable properties of cement-based waste form such as porosity and leachability. Diffusion coefficients and leach indecies of coated specimens were calculated and show acceptable records.

It could be stated that coating cemented waste form by bitumen emulsion, isolate the radioactive contaminants, thus reduces their back release to surrounding and in consequently save the environment proper and safe.

Key words: Solidification/stabilization, coating, cement, bitumen, radioactive waste, porosity, leachability

INTRODUCTION

Solidification/stabilization (s/s) has a very important role in handling, transportation, interim storage, and final disposal of radioactive wastes. For safety, the release of hazardous radioactive materials from the disposal site into the environment should be prevented or delayed.

Cement and bitumen have been used as immobilization matrices for the radioactive wastes from the beginning of the nuclear industry in the 1960's [(1)].

The durable cement materials are currently the preferred one for embedding of most of low and intermediate level radioactive wastes due to the acceptable strength obtained for the final waste form and hence could be handled, transported and disposed safely [(2-3)].

Bituminized waste forms resist the water penetration exhibiting very low leachability of contaminants because of its impervious matrix and smooth surface. Bituminization, accompanied by both mixing and evaporation processes, can lead to high volume reduction. In addition, bitumen has good coating properties resistant to attack by microorganisms, consequently, more optimal characterization of the immobilized waste form was achieved. However, bituminized waste forms, prepared by using especially straight-run distillation bitumen, are mechanically and dimensionally unstable [(4-6)]. This instability is closely related to the long-term mechanical stability of waste forms at the disposal site.

The aim of this study was to obtain waste form with sufficient strength and high mechanical integrity of cement materials and, in the same time, with relatively high leaching stability, low fractureability and more elasticity.

In the present work simulated cement waste forms were prepared by mixing water with cement then coating the obtained blocks with bitumen emulsion by immersion process. Mechanical characterizations and porosity of the obtained products were evaluated under different experimental conditions. Beside, X-ray diffraction, infrared and electron microscope investigations for the final waste forms were performed. The chemical stability of the coated waste forms was compared with the non-coated ones to evaluate the leachability of radionuclides in different leachants.

MATERIALS AND METHODS

1. Materials:

Cement materials: Portland cement (PC) CEM1 (N/42.5) used in the present work was the available local cement manufactured according to Egyptian Standard Specifications ES 4756-1/2005 and EN 197-1/2004 (7). The chemical analysis of the cement type as well as the Bogue calculation (8) for its four major compounds is given in table (1).

Table (1): Chemical composition (wt.%) and compounds compositions (%) of the cement sample used

Chemical composition, %		*Compounds composition, %*	
SiO_2	19.84	**C_3S**	53.11
Al_2O_3	4.74		
Fe_2O_3	4.0	**C_2S**	16.89
CaO	61.01		
MgO	2.5	**C_3A**	5.81
K_2O	0.6		
SO_3	2.4	**C_4AF**	12.16
Insoluble residue	0.95		

-Loss on ignition = 3.96%.

-Lime saturation factor = 96%by wt.

Bitumen: The bitumen used in this study for the coating process was commercially available straight-run distillation bitumen, having the elemental analysis described in table (2).

Table (2): Elemental analysis of the bitumen used in coating, (%)

Carbon (%)	*Hydrogen (%)*	*Sulphur (%)*	*Nitrogen (%)*
89.34	7.07	0.140	0.178

Leaching solutions:

Three types of water, namely tap, ground and sea water, were used in this study either in leaching experiments or for immersion tests.

The tap water used was the normal tap water in Giza district and was used for sake of comparison with the other two types of leachants. The ground water was obtained from Abu-Zaabel well (No.202) which is one of the nearest ground water wells to Inshas-Reactor site. Since the sea water in the world has mainly similar composition, therefore, sea water was obtained from Alexandria, on the Mediterranean Sea. The concentration of some ions of interest in the three leachants used is shown in table (3).

Table (3): Chemical analysis of some ions of interest in different types of leachants

Leachants	*pH*	*Soluble cations (ppm)*				*Soluble anions (ppm)*		
		K^+	Na^+	Mg^{2+}	Ca^{2+}	Cl^-	SO_4^{2-}	HCO_3^-
Tap water	6.90	0.086	1.07	1.2	1.4	0.77	0.7	1.8
Ground water	7.20	23	149	13	74	137	317	272
Sea water	7.93	8.4	652.6	96.9	28.06	496.9	60.8	183

ppm ≡ mg/l

Radioactive isotopes: Cesium-137 and cobalt-60 are two of the most famous radionuclides found in numerous types of low and intermediate level radioactive wastes and are used in this study to trace the leaching characterization of the final coated waste forms.

2. Experimental Methods:

2.1- Preparation of coated cement form:

Cylindrical blocks of cement materials were prepared by mixing cement powder with water at the 0.35 w/c ratio and poured into a cylindrical plastic mold till hardening. For leaching experiments radioactive water solution (Cs-137 + Co-60 ≈ 831Bq/ml) was used for producing blocks subjected to chemical resistance tests. After 28 days solid cemented waste forms having dimensions of 60±2 mm height and 31±0.5 mm diameters were obtained. The cement blocks, at the end of curing time, were immersed in soft hot bitumen at 90^oC then left out to be cold and hardened for one week before performing the different investigating tests. There was an increase of 10±1% in the weight of waste form due to coating process, related to the original weight of the blocks before coating.

Fig. (1) represents the photo image of cement waste forms (a), before and (b), after coating process.

Fig. (1): Photo image of non-coated cement block (a) and that coated with bitumen emulsion (b)

2.2- Characterizations of the final waste forms:

a- Compressive strength: Mechanical integrity was studied by measuring the compressive strength of solid cement blocks and the coated ones. The compressive load required to cause damage were determined for 3-5 cylindrical blocks. A hydraulic press from Ma test, (Italy), model, E 159 SP was used for this test.

b- Immersion test: Immersion test was carried out for non-coated and the coated cement blocks by immersion for 90 days in tap, ground and sea water. Visual examinations, compressive strength as well as the weight of solid blocks were measured periodically.

c- Porosity test: According to ASTM C-642 saturation technique, the permeable porosity of the samples, applying the cold water saturation method, (CWS) was determined and calculated according to the following equation [(9-11)].

$$\text{Porosity (\%)} = \frac{\text{wet mass (g) - dry mass (g)}}{\text{volume of the block (cm}^3\text{)}} \times 100$$

d- Freezing-thawing test: Freeze-thaw cycling test was performed for solid samples of non-coated cement blocks and coated groups. The resulting free standing coated and non-coated blocks after 28 days were subjected to successive cooling and heating cycles every 24 hours in a fridge at $-50\,^oC$ and then in an oven at $60\,^oC$ up to three months period. Mechanical integrity and weight changes due to freeze thaw were evaluated.

e- Leaching test: The chemical resistances of the coated and non-coated solid cement blocks were evaluated by following the leaching of radionuclides from specimens immersed in different leachants. Leaching tests were performed according to the method recommended by the International Atomic Energy Agency, [(12)]. The only difference is that the whole solid blocks were immersed completely in the leachant solutions. The radionuclides content of the leachates were measured using multichannel analyzer, PCAP, USA.

f- Spectroscopic analyses: FT-IR analyses were carried out by FT-IR - Jasco, 460 plus equipment, spectra and were usually recorded in the range of 4000–400 cm^{-1} with 2 cm^{-1} resolution. On the other hand, X-RD analyses were carried out with a Philips Analytical X-Ray B.V. operating with Cu tube anode.

g- Electron microscope: Jeol JXA-840A Electron Probe Microanalyzer scanning electron microscope (Japan) was employed with stage magnification of x 2,500. The actual pretreatment of preparation consisted only of gold plating of the samples in S150A Sputter Coater Edwards (England) before examination.

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RESULTS AND DISCUSSION

Bitumen is virtually impermeable to water and has very low solubilities and compatible with most environmental conditions. Therefore, coating the cemented waste form reflected these stated advantages beside that inherited from cement, so as the necessity for coating process was appeared.

1- Compressive strength and porosity properties:

Quality of Portland cement (PC) aggregate, such as shape and surface roughness, is one of degree of porosity factors [13]. Table (4) showed the test results of the compressive strength of control PC when mixed with different water/cement (w/c) ratios from 0.25 up to 0.50 (wt/wt). It is clear that the compressive strength increases gradually till 0.35 w/c ratio due to completing the hydration reaction with increasing water. Then mechanical integrity decreases again gradually with increasing w/c ratio according to the excess of water. Therefore, the optimum w/c ratio is at 0.35, which is similar trend with that recorded in the literature [14].

Table (4): Compressive strength of the cement waste form after coating process with different water/cement (w/c) ratios

Water/cement (w/c) ratio (wt/wt)	*Compressive strength (MPa)*
0.25	38.97
0.30	41.72
0.35	44.79
0.40	39.47
0.45	35.9
0.50	32.07

In this study, the high of porosity percentages for Portland cement blocks rough surface were decreased from 31.5±.07 % to 9.7±1.1 %, while the compressive strength values decreased in case of coated blocks (from 44.79 MPa for cement waste form at 0.35 w/c to 23.15 MPa for coated ones). This may be due to the elasticity inherited by the waste form after the coating process with the bitumen. The cement blocks were flowed rather than fractured. This consider as advantages by avoiding the dispersion of the hazardous components to the environment in case of their damage.

Immersion in different types of water:

The immersed coated and non-coated cement blocks in three different types of water were followed for time intervals of 30, 60 and 90 days to evaluate the long-term stability of the final waste forms. The compressive strength values for each group at the end of each interval were measured and the data reached are shown in table (5). The main trend indicate that, the compressive strength values of coated and non-coated cemented waste forms decreased by progress in immersion time for 30 days in the three different waters tested. The mechanical integrities increased again after 60 and 90 days of immersion periods. It is worth mentioning that these values do not exceed those values of unimmersed groups at the same time intervals. The first decrease in compressive strength of coated and non-coated cement blocks may be due to the diffusion of water into the pores of the specimens and hence, induce the release of lime and calcium sulphate components may occur which leads to lose in the mechanical strength of the blocks [15]. While the second increase in compressive strength after 60 and 90 days could be contributed to the carbonation process occurred, where the calcite claim to be formed due to the reaction of $Ca(OH)_2$ of cement and CO_2 of surrounding environment and fill the pores of immersed blocks [16].

Table (5): The compressive strength values of coated and non-coated cement blocks after immersion in different water

Immersion time, (days)	*Compressive strength (MPa)*					
	Tap water		**Ground water**		**Sea water**	
	non-coated	**coated**	**non-coated**	**coated**	**non-coated**	**coated**
0	44.79	21.4	44.79	21.4	44.79	21.4
30	31	18.7	29.0	23.3	27.4	18.6
60	32.5	27.7	30.8	25.1	30.87	23.05
90	35.4	28.8	32.8	26.2	33.1	24.8

Figure (2) describe the change in weights of non-coated cement blocks which increased by immersion in sea water with more than 6% compared to that in tap and ground water where 4% increase in weight was only recorded. This may be due to the salinity of the sea water which leads to accumulation of more salt layers on the block surface compared to that immersed in the other two waters. On the other hand, low permeability of bitumen layer is related to low penetration of water into coated cement blocks causing undetectable weight increases in the specimens (not exceed 1%).

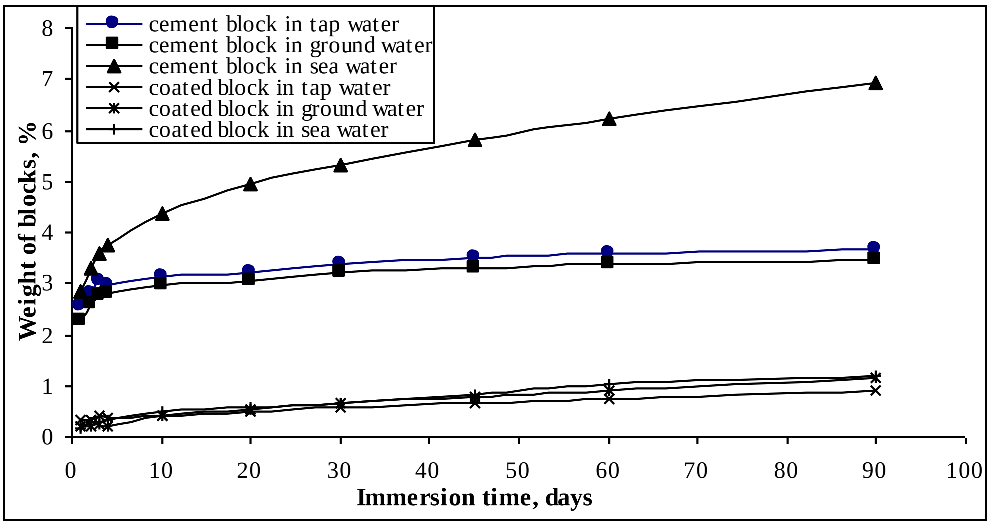

Fig. (2): Change in weight by immersion of coated and non-coated blocks in different waters with time.

Freezing Thawing test:

Table (6) represents the data obtained for the freezing thawing test on both non-coated and coated blocks after 7, 14, 30 and 90 cycles intervals. Decrease in the compressive strength for coated sample was recorded and reached 8.7 MPa after 90 freezing-thawing cycles, compared to 22.59 MPa lose in the mechanical integrity of non-coated samples, table (6). This indicated that coating the cement waste form by bitumen enhances their resistance to the disposal site climatic changes.

Table (6): Compressive strength values for non-coated and coated cement blocks subjected to freezing thawing treatment

Freezing thawing cycles	*Compressive strength, (MPa)*	
	non-coated	**coated**
0	44.79	21.4
7	33.42	18.42
14	29.1	16.91
30	29.1	14.6
90	18.2	12.7

Chemical characterization:

The mathematical theory of transport by diffusion from solid, based on Fick's law, can be used as analytical solution for the leaching of radionuclides from waste forms [17-18].

Cumulative leach fraction of radioactivity released is derived as:

$$\sum a_n/A_o = 2(S/V)\,(D_e\,.\,t/\pi)^{1/2}$$ Where:

$\sum a_n$ =Total amount of radioactivity released in all leaching periods up to time, t

A_o = Initial amount of radioactivity of interest originally present

S = Specimen surface area, cm^2

V = Specimen volume, cm^3

D_e =Effective diffusion coefficient

Also, the leachability index (Lx), as a figure of merit [19], can be calculated using the following equation:

$$Lx_i = \frac{1}{i} \sum_{n=1}^{i} \log\left(\frac{1}{D_e}\right)$$

Where i = the number of leaching periods.

When the cemented waste forms come in contact with water, the hazardous soluble materials, including the radionuclides, moves from the waste form to the surrounding water and this due to dissolution or chemical reaction with the components of water. Therefore, maintaining stable chemical and physical properties of waste form is very important task to delay or even to prevent the release of hazardous radionuclides into the environment when it comes in contact with water in disposal site.

Bitumen is chemically inert matrix, insoluble in water and show a remarkable lower leach rate of the final waste form when compared to the cement products.

Fig. (3) represents the cumulative fractions of radionuclides leached from coated and non-coated waste form specimens as a function of time.

It is clear from figure that the leachability after coating process show reduction up to 90% compared to non-coated specimens. These figures are in a good agreement with the work published by Matsuzuru [20]. This could be explained in view of low porous surface of coated specimens which retarded break throw of water into cement block specimen and consequently delayed the radionuclides release [21]. This could be compared to the non-coated porous cement which shows more water penetration into the specimens. In addition, it is clear from Fig. (3) that there is no significant differences in cumulative leach fractions for coated specimens when exposed to the three leachants. On the other hand, in case of non-coated specimens, the releasing of radionuclides into sea water is higher than that of tap and ground water. This may be attributed to the high salinity and the high concentration of soluble ions in sea water which enhance the exchange of radionuclides of non-coated specimens compared to that in ground or tap water.

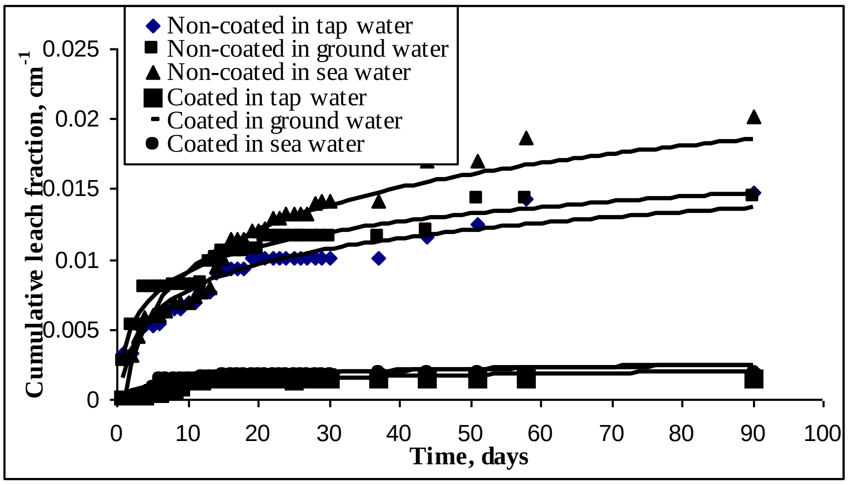

Fig.(3): Cumulative leach fraction for coated and non-coated cement specimens versus leaching time

According to the equation of the average effective diffusion coefficient (D_e) and leachability index (Lx), the values of D_e increases while Lx decreases and the data are reported in Table (7). The higher leachability index for coated specimens compared to non-coated ones confirming the chemical improvement obtained by coating process.

Table (7): Effective diffusion coefficient (D_e) and Leachability index (Lx) of radionuclides for coated and non-coated waste forms

Leachant solution	*Non-coated*		*Coated*	
	D_e	**Lx**	**D_e**	**Lx**
Tap water	1.23E-11	9.217728	2.06E-13	8.708159
Ground water	1.71E-11	9.052412	3.6E-13	10.51706
Sea water	1.65E-11	9.165989	3.61E-13	10.71301

Microscopic examination:

Figure (4) shows the scanning electron micrographs of non-coated cement waste form and the coated one. It is clear from photo (4-a) the rough surface of hydrated cement specimens. On the other hand, the coated block in photo (4-b) appears as smooth surface. This figure also illustrate the role of bitumen emulsion in coating the hydrated cement and entering into its pores giving less permeable waste form and consequently an acceptable improvement in the chemical criterion as reached from the chemical test data.

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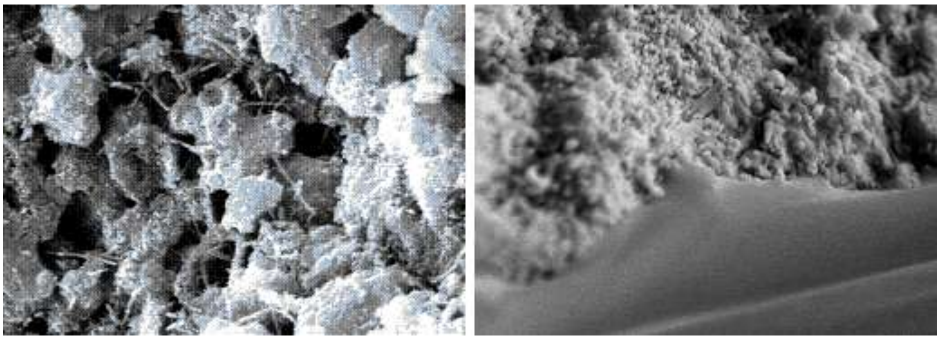

(a) **(b)**

Fig. (4): The scanning electron micrographs of fracture surface of Portland cement waste form (a) and coated one with bitumen matrix (b)

Infrared spectroscopy:

The infrared analysis was performed for coated and non-coated cement blocks after 28days of molding and gave nearly identical peaks assignment for the two samples as shown in fig. (5). Hydrated cement is assigned by more known absorption bands such as a shoulder band at 3642 cm^{-1} which related to free water molecules in vapor phase and a broad band at 3450cm^{-1} which is due to symmetric and asymmetric stretching vibration of adsorbed water molecules. Also, the peak at 1650cm^{-1} may be accord to the deformation vibration of water molecules in cement. $Ca(OH)_2$ and $CaCO_3$ which produced during setting and curing of cement in presence of atmospheric carbon dioxide, may be represented by the band at 1425cm^{-1}.

The band of about 1000cm^{-1} is identical for C-H-O and that at the vicinity of 875cm^{-1} may be attributed to stretching vibration of Si-O in hydrated cement, Fig. (5). These data are in agreement with the previous reported works [(22-24)]. The peaks assignment for coated and non-coated cement blocks are typically the same as previously stated, so coating with bitumen has no effect on the functional groups and hence the cement preserve cement blocks their good mechanical integrities.

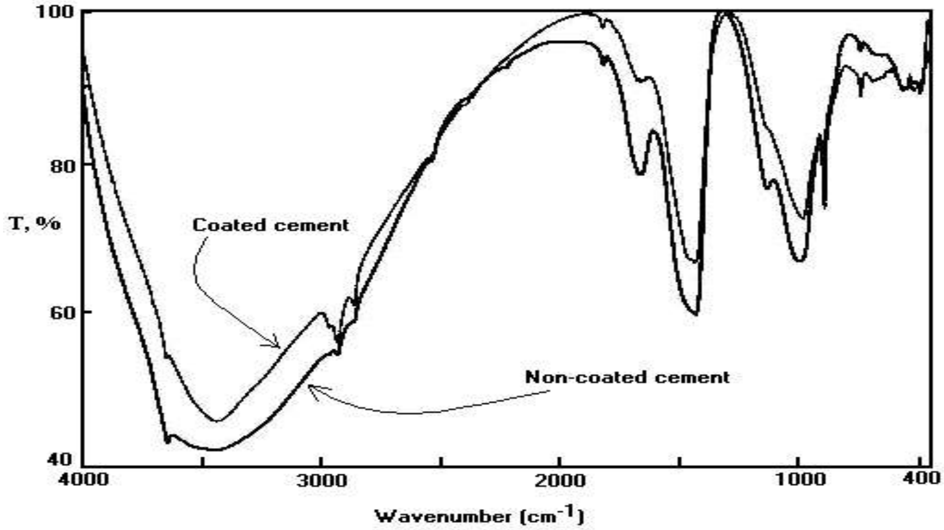

Fig. (5) IR spectra of coated and non-coated cement waste form

X-Ray Diffraction:

Fig. (6) represent d-values for portlandite $Ca(OH)_2$ and calcite $CaCO_3$ in coated and non-coated cement blocks. It is worth mentioning that d-values are typically the same with some difference in relative intensity percent. The obtained records are in a good agreement with those reported in references for portlandite and calcite [(25)]. This indicated that coating cement blocks with bitumen matrix did not affect negatively on the cement waste form. The same conclusion was reached from the infrared analysis.

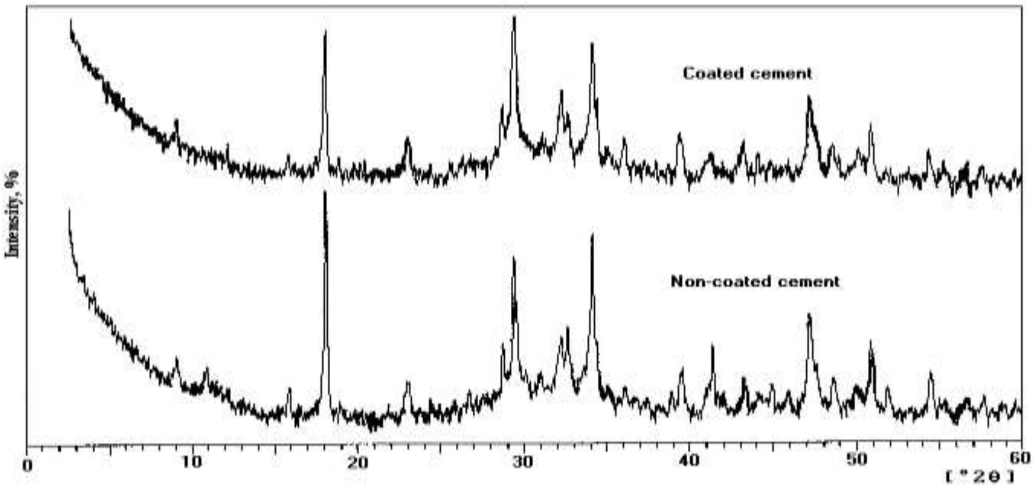

Fig (6): X-RD of coated and non-coated cement waste form

CONCLUSION

The experimental results indicated that coating the simulated cement waste form by bitumen emulsion improved their porosity, resistance to immersion and freezing thawing and chemical stabilities. In addition, leachability of simulated cement waste form was lowered to about 90% of its value by performing bitumen coating process.

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التغطيه بالبتومين كوسيلة لتحسين المسامية والثبات الكيميائى لمماثل النفايات المصلدة بالأسمنت

حسام الدين مصطفى صالح

قسم النظائر المشعة – مركز البحوث النووية- هيئة الطاقة الذرية المصرية

تم فى هذا البحث دراسة كيفية تغطية المنتج النهائى لمماثل النفايات المشعة الأسمنتى الصلب بالبتومين و تم بعد ذلك إجراء اختبارات للخواص الكيميائية والطبيعية للمنتج المغلف على المستوى المعملى. كما تم استخدام كلا من حيود الأشعة السينية والأشعة تحت الحمراء بالإضافة الى التصوير بالميكروسكوب الإلكترونى لفحص عينات مغلفة وأخرى غير مغلفة للمنتج النهائى الأسمنتى الصلب.

وقد أوضحت النتائج التى تم التوصل اليها أن عملية التغليف أو التغطية للمنتج النهائى الأسمنتى الصلب لمماثل النفايات المشعة بالبتومين قد أدت الى تقليل المسامية وبالتالى زيادة الثبات الكيميائى للمنتج. ويمكن القول أن عملية التغطية للمنتج الأسمنتى بالبتومين تعزل الملوثات المشعة داخل المنتج الصلب وتقلل أو قد تمنع تسربها للوسط المحيط ونتيجة لذلك يتم الحفاظ على البيئة سليمة وأمنة.