Initial studies on the effects of radiation, thermal ageing and aqueous environments on the stability and structure of candidate polymeric encapsulant materials

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ABSTRACT

The current route in the UK for the conditioning and immobilization of most intermediate level waste for interim storage and geological disposal is to encapsulate in a cementitious matrix. However, certain waste materials, such as those containing reactive metals (e.g. uranium and aluminium), can corrode in the presence of the highly alkaline water in a cementitious environment. In their initial, undegraded form, polymeric materials can provide the appropriate, unreactive environment needed for the encapsulation of chemically active metals.

This study examines the effects of gamma radiation on the stability of six candidate polymeric encapsulants, including a vinyl ester styrene resin (VES) and five epoxy resin formulations. The polymeric encapsulants were exposed to radiation doses up to 10 MGy using AMEC's cobalt-60 gamma irradiation facility and their radiation and chemical stability characterized by the use of a number of analytical techniques. These included flexural and compressive testing, Fourier transform infrared spectroscopy (FTIR), gel fraction, leach testing and gas evolution. The results show that the most stable resin in terms of radiation resistance and chemical stability was VES. Most of the epoxy resin materials also showed good generic stability, but the FTIR analysis showed the potential for dose-rate effects in one formulation.

KEYWORDS: polymeric encapsulants, radiation, dose-rate effects.

Introduction

THE current route in the UK for the conditioning and immobilization of most intermediate level waste during interim storage and geological disposal is to encapsulate in a cementitious matrix. However, certain radioactive waste materials, such as those containing reactive metals (e.g. uranium and aluminium), can corrode in the presence of the highly alkaline water in a cementitious environment. In their initial, undegraded form, polymeric materials can provide the appropriate, unreactive environment needed for the encapsulation of chemically reactive metals. A potential drawback to the use of polymers as encapsulants for radioactive materials is that they can be susceptible to degradation when exposed to ionizing radiation. Consequently, research has been initiated by the Nuclear Decommissioning Authority, Radioactive Waste Management Directorate (NDA RWMD) and Sellafield Ltd to study the effects of gamma irradiation on a range of candidate organic polymeric encapsulants.

The changes in chemical structure that occur when organic polymers are exposed to ionizing radiation are a consequence of ionization and free radical processes. The extent of the radiation induced changes is dependent on many factors

such as the irradiation environment, temperature, dose rate and the presence of additives. In particular, the radiation induced changes can become significant if oxygen is present. Interactions between free radicals and oxygen leads to the formation of aggressive peroxide radicals which induce degradation by oxidative chain scission.

Ideally, radiation resistance assessments of the candidate encapsulant materials would be carried out under conditions identical to those expected in long-term storage where radiation dose rates are expected to be low, but doses of 10 MGy might be accumulated over many years. Clearly it is not possible to replicate fully these long exposure times in normal experimental timescales. Therefore the radiation testing in this study was accelerated and carried out at high dose rates to achieve a dose of 10 MGy within acceptable timescales. Some additional irradiations at low dose rates were also carried out by Dawson *et al.* (2010).

Experimental methods

Materials

All the polymer encapsulants characterized in this study are commercially available and are described as follows. (1) Two APS (advanced polymer system) epoxy resins: one a standard APS formulation and the other low-temperature APS formulation (LT-APS), which has been achieved by altering component mix ratio. These resins have been sourced through Diversified Technologies Services in the USA. (2) Two epoxy resins formulated by Alchemy UK: one a standard formulation called Alchemix 4760 and the other a low temperature version of that formulation (LT-ALC), which has also been achieved by altering component mix ratio. (3) An epoxy resin produced by Huntsman in the UK obtained by mixing 100 parts of Huntsman LY 1564 Bisphenol A resin with 35 parts Huntsman XB 3403 polyetheramine hardener. (4) A vinyl ester styrene (VES) formulation, known as Dow Polymer originally manufactured by the Dow Chemical Company used in the VERI (vinyl ester resin *in situ*) encapsulation process (Diversified Technologies, 2000).

Mechanical testing

The effects of gamma irradiation on the structural integrity of the epoxy resins and the VES were evaluated using flexural and compressive testing. The sample dimensions for the flexural tests were approximately 80 mm length, 15 mm width and 4.8 mm thickness and approximately 10 mm length, 10 mm wide and 4.8 mm thickness for the compressive tests.

Batches of twelve three-point bend samples and twelve compressive samples of each of the standard APS and Huntsman resin formulations were irradiated in air at room temperature. All irradiations were carried out in AMEC's cobalt-60 gamma irradiation facility in the UK, this facility has the capability of irradiating materials and components over a wide range of dose rates and a wide range of conditions (e.g. elevated temperatures and in corrosive process liquors). Due to sample availability, the batches of Alchemix and VES contained only six samples. Batches were irradiated at a dose rate of 4 kGy hr^{-1} to a dose of 150 kGy and a higher dose of 10 MGy. Sample batches from the low temperature versions of the APS and Alchemix formulations were only irradiated at ~4 kGy hr^{-1} to a total dose of 10 MGy to serve as spot check comparisons with the standard formulations.

During the flexural tests, samples were mounted in a three point bend fixture and tested to failure on an Instron 1195 tensile test machine using *Instron Series IX* analysis software. All samples were tested at a crosshead speed of 1 mm min^{-1}. The sample span of the three-point bend fixture was 37 mm. During compression testing, samples were placed between steel platens separated by 10 mm on the same test machine. The samples were then compressed to failure at a crosshead speed of 1 mm min^{-1}.

Fourier transform infrared spectroscopy

Samples that were used for flexural testing were characterized using Fourier transform infrared spectroscopy (FTIR) on a Varian Digilab Excalibur spectrometer fitted with a UMA 600 FTIR microscope. The outer edge and middle of the cross-sections (i.e. the central zone) of the freshly exposed fracture surfaces were analysed using a permanently aligned attenuated total internal reflection (ATR) attachment. The broken flexural test samples that were used for this analysis were broken again immediately before FTIR analysis to generate freshly exposed fracture surfaces for each sample.

It should be noted that all of the epoxy spectra were normalized so that the peak heights of the p-

phenylene peak at ~830 cm^{-1} were consistent in each case so that any differences arising from inhomogeneity in the samples was minimized. The peak at 830 cm^{-1} was also selected for VES, but corresponds to a C–H stretch along the polymer backbone.

Gel-fraction and solvent uptake factor

Gel fraction and solvent uptake factor measurements were carried out on samples of approximately 100 mg from each of the epoxy resin and VES samples that were irradiated to 150 kGy and 10 MGy. Measurements were also carried out on unirradiated samples to act as controls.

Each of the samples were carefully weighed to establish initial weights, w_o, and then the epoxy resin samples were refluxed in tetrahydrofuran (THF) for 24 h. The VES samples were refluxed in dichloromethane for the same time period. After refluxing, the samples, swollen with solvent, were quickly transferred to clean glass vials of known weight to trap any volatile solvent. The combined vial and sample weights were recorded and the weight of the swollen samples, w_s, calculated. The samples were then allowed to dry under vacuum until their weights stabilized and their final weights, w_f, were calculated. The gel-fraction is given by the ratio of w_f to w_o and the solvent uptake ratio is defined as the ratio of w_s to w_f.

Leaching characteristics

Chemical stability

The leaching characteristics of the encapsulants were investigated by placing a known mass of each material in a stoppered glass vessel containing either deionized water (pH 7) or calcium hydroxide solution (pH 12.4). Testing of each resin was carried out in duplicate and were either aged at room temperature or thermally aged at 80ºC. All experiments were set up within a glovebox, under a nitrogen atmosphere. The Ca(OH)$_2$ solutions were kept saturated by maintaining a slight excess of solid Ca(OH)$_2$ in the test vessels, thus ensuring a steady pH of ~12.4. During the ageing period, leachates were periodically taken for total organic carbon (TOC). Once ageing was complete, leachates were also sent for organic screening tests using gas chromatography-mass spectrometry (GC-MS) and ion chromatography.

Radiation stability in aqueous solution

The sample preparation and sampling procedures for the irradiated samples was the same as described for the chemical stability leaching tests except that the atmosphere above the solutions was air and samples were only aged at room temperature. The samples were irradiated to a total dose of 10 MGy at 3.2 kGy hr^{-1}.

Gas analysis

Gas samples were prepared by weighing ~1 g of small resin particles (~1 mm^3 in volume) into glass tubes. A glass blower then sealed the glass tubes to form cylindrical glass ampoules of approximately 3 cm^3 volume. All of the resins were encapsulated in air and a protracted weak spot was left at the sealing point in each ampoule for ease of breaking before eventual gas analysis.

The glass ampoules containing resin were irradiated to 150 kGy and 10 MGy at 4 kGy hr^{-1}. The LT-APS and LT-ALC resin samples were only irradiated at 4 kGy hr^{-1} to a dose of 10 MGy to act as spot checks for comparison with the standard formulations.

The irradiated and control ampoules were placed within larger standard gas analysis bottles (referred to as head space vials) in an argon filled glove-box. The headspace vials were then sealed with composite aluminium/PTFE/silicone septa such that the ampoules were sealed in argon gas. Just before gas analysis, each headspace vial was carefully shaken until the protracted weak spot in the sealed ampoule broke releasing gas into the inert atmosphere in the headspace vial.

Results and discussion

Visual appearance

Figure 1 shows that after irradiation ageing to 10 MGy all polymers become darker as a result of radiation induced changes to the optical absorption properties.

Flexural and compressive test samples

The flexural strength of a sample in three point bend (the maximum flexural stress reached in a test), can be calculated using the following formula (Lever and Rhys, 1968):

$$\sigma_f = \frac{3LF}{2bd^2} \qquad (1)$$

FIG. 1. Photographs of some of the candidate encapsulants before (top) and after irradiation to 10 MGy (bottom). From left to right: APS, Alchemix, Huntsman and VES.

where σ_f is the flexural strength; L is the test fixture span in m; F is the force in N; b is the sample width in m; and d is the sample thickness in m. The units of flexural strength are MPa.

Table 1 shows the results of the flexural and compressive testing for all candidate resins. The data show that irradiation to 150 kGy produced little or no change in the flexural strength in any of the candidate resins but after a dose of 10 MGy, all the epoxy resin samples showed a reduction in flexural strength. However, no significant change in the flexural strength was observed for the VES at 10 MGy.

The flexural strength of the original APS formulation reduced by 75% and the LT-APS reduced by 65%, suggesting that the LT version could have a higher radiation tolerance. Although the Alchemix showed slightly better radiation tolerance than the APS, the reduction in the flexural strength of the Alchemix (47%) and the LT-ALC (32%) followed a similar trend to that of the APS. However it should be realized during these comparisons that the flexural strengths of the unirradiated low cure versions of the APS and Alchemix were lower than those of the original respective formulations.

The compressive data for the Alchemix, LT-ALC, APS and LT-APS resins show that although gamma irradiation up to 10 MGy reduced their flexural strength, irradiation to

TABLE 1. Results of the flexural and compressive strength testing after gamma irradiation to the stated doses at a dose rate of 4 kGy hr^{-1}.

Resin type	Total gamma dose	Flexural strength (MPa)	Mean compressive yield stress (MPa)	Mean stress at failure (MPa)
APS	0	35.4(1.4)	21.2(1.0)	81.8(8.0)
	150 kGy	35.9(1.4)	23.7(1.4)	111.5(20.6)
	10 MGy	8.3(1.9)	22.8(2.5)	109.4(12.9)
LT-APS	0	24.8(1.1)	15.9(0.6)	96.8(9.9)
	10 MGy	8.5(3.0)	16.4(0.97)	102.3(9.1)
Alchemix	0	112.9(0.9)	82.8(3.5)	116.2(14.3)
	150 kGy	114.7(1.8)	79.4(1.1)	119.9(22.8)
	10 MGy	69.3(3.7)	86.0(3.2)	92.1(9.0)
LT-ALC	0	78.2(3.2)	83.8(6.4)	200.5(21.3)
10 MGy	42.4(11.1)	89.6(4.8)	145.8(22.0)	
Huntsman	0	97.6(2.9)	62.7(3.6)	216.2(33.2)
	10 MGy	No test possible	No test possible	No test possible
VES	0	107.8(4.3)	78.9(3.5)	122.9(46)
	150 kGy	118.3(9.2)	87.0(5.4)	166.6(66.1)
	10 MGy	112.4(13.8)	90.5(7.5)	106.7(24.3)

Figures in parentheses correspond to the standard deviation.

2988

this dose did not significantly affect their compressive strengths.

The data generated for the Alchemix and APS epoxy resins and VES after ageing to 10 MGy suggest that these materials show good radiation resistance when tested in compression. However, the data also show that at the same radiation dose the application of a tensile stress component during flexural testing to the epoxy resins leads to a reduction in mechanical properties and consequently the radiation stability of the epoxy resins at 10 MGy is not as good as might be concluded from compressive data.

The degradation in the Huntsman samples at 10 MGy was so severe that it was not possible to carry out any flexural or compressive tests. The irradiation caused the samples to lose their structural integrity such that they behaved more like a 'mastic' or a putty rather than an epoxy resin.

Fourier transform infrared spectroscopy

There is a generic similarity between the FTIR spectra of the unirradiated epoxy based encapsulants, although there are differences in the spectra at ~1040 and ~2900 cm^{-1}. Table 2 gives the

TABLE 2. Assignments of peaks in epoxy resin FTIR spectra.

Wavenumber (cm^{-1})	Assignment
3300	NH and OH stretch
2925, 2967	CH$_2$, CH$_3$ asymmetric stretch
2885, 2872	CH$_2$, CH$_3$ symmetric stretch
1740	C=O, carbonyl stretch
1605, 1580, 1510	Ring −C=C−H stretch
1452, 1375	Methylene CH$_2$, CH$_3$ bend
1260, 1180	Ring−O stretch
1161, 1033	C−O−C ether linkages
1100	−O−C−C stretch
1109, 1046	−C−N
915 (950−810)	CH$_2$−O−CH epoxy bend
829, 773	Ring =C−H, C−H

suggested assignments for the main peaks observed in the spectra for these resins (Fig. 2 shows example spectra).

The APS and LT-APS spectra shown in Fig. 2 compare the outer edges and interior of samples irradiated to 10 MGy with the spectra of an unirradiated sample. The LT-APS spectra show
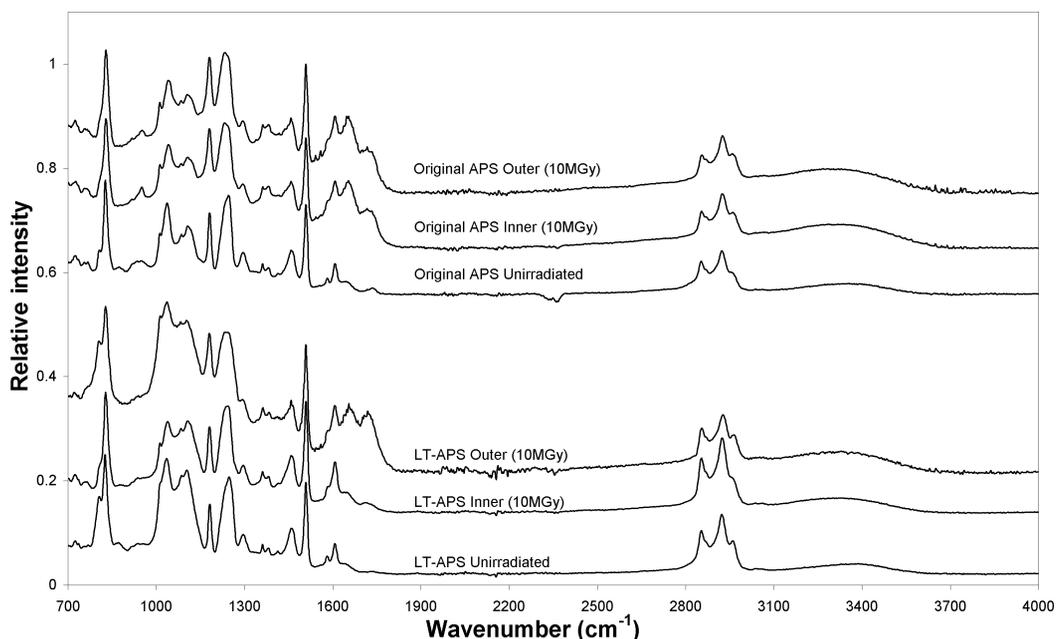

FIG. 2. Comparison of the FTIR spectra of APS and LT-APS before and after irradiation to 10 MGy. Spectra taken from the central zone and outer edge of the irradiated samples are also compared.

2989

that the intensity of the bands at 1650 and 1740 cm^{-1} are significantly lower in the centre of the sample relative to the outer edge. This observation suggests that the outermost edges of the sample were more heavily oxidized than the interior. This effect was also observed for LT-ALC, but to a lesser extent.

The VES spectra are shown in Fig. 3 and show that after irradiation to 10 MGy, there was a significant decrease in intensity of the bands at ~960 and 1300 cm^{-1}, and a slight decrease in the bands at ~3000 cm^{-1} which are most probably associated with C−H stretches. There was also a slight reduction in intensity and broadening of the band at ~1700 cm^{-1} assigned as a carbonyl stretch.

Gel-fraction and solvent uptake factor

Gel-fraction is a useful tool for characterizing the amount of cross-linking that is present in a polymer. In general, for irradiated polymers, when cross-linking dominates, the percentage gel-fraction increases and the solvent uptake factor decreases. If chain scission dominates, however, the opposite trends tend to occur.

The gel-fraction and solvent uptake data for the irradiated epoxy resins and VES are shown in Table 3. The gel fraction data for the APS remained constant at about 0.93 after irradiation to 150 kGy, but decreased to 0.75 at 10 MGy. Corresponding increases in solvent uptake ratio were also observed at 10 MGy. The data suggest that irradiation to 150 kGy had little effect on the

APS but on irradiation to 10 MGy, the decrease in gel-fraction, increase in solvent uptake factor and change in flexural strength are consistent with chain scission at this dose.

In both Alchemix formulations, however, increases in gel-fraction and corresponding decreases in solvent uptake values were observed after irradiation to 150 kGy suggesting that some cross-linking had occurred at this dose. At 10 MGy, the gel-fraction values decreased for each of the Alchemix formulations and the solvent uptake factors increased. These observations, coupled with a reduction in flexural strength, are consistent with those expected for a material that has undergone chain scission.

In the Huntsman resin, no effective difference was observed in gel-fraction or solvent uptake factor at 150 kGy. At 10 MGy, however, the heavily degraded irradiated resin dissolved completely, so no measurements could be carried out.

The VES resin showed similar changes to those observed for APS and Alchemix where an increase in gel fraction at 150 kGy was then accompanied by a decrease in gel fraction and an increase in the solvent uptake factor at 10 MGy. This was consistent with the onset of chain scission in VES at this dose.

Gas analysis

Figure 4 shows that the gas evolution for all of the epoxy resin formulations irradiated to 10 MGy in air was dominated by the production of hydrogen.
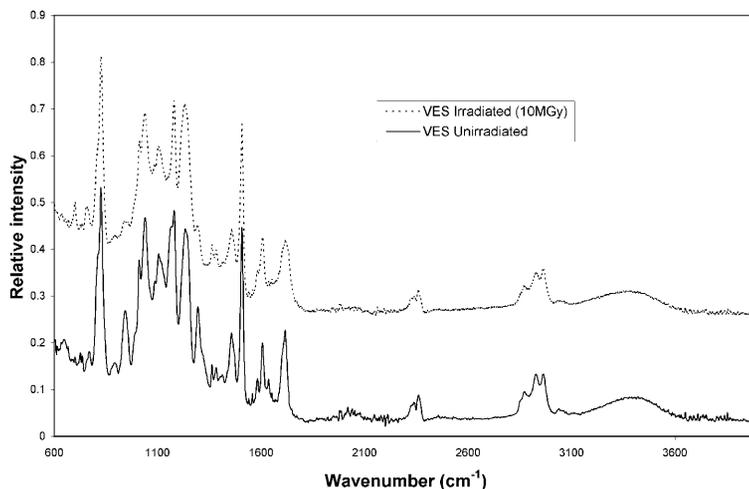

FIG. 3. The FTIR spectra of VES before and after irradiation to 10 MGy.

TABLE 3. Solvent uptake factor and gel fraction data for the candidate polymer encapsulants after gamma irradiation at a dose rate of 4 kGy hr^{-1}.

Resin type	Gamma dose	Solvent uptake factor	Gel fraction
APS	0	1.95	0.93
	150 kGy	2.06	0.92
	10 MGy	3.34	0.76
LT-APS	0	1.79	0.94
	10 MGy	2.33	0.83
Alchemix	0	1.94	0.94
	150 kGy	1.91	1.00
	10 MGy	2.35	0.94
LT-ALC	0	1.86	0.94
	150 kGy	1.83	0.99
	10 MGy	2.15	0.92
Huntsman	0	1.86	1.00
	150 kGy	1.86	1.00
	10 MGy	No test possible	
VES	0	1.56	0.95
	150 kGy	1.62	0.99
	10 MGy	2.03	0.93

The graph also shows that under the same ageing conditions, the gas evolution data for VES were different to those observed for the epoxy based materials. The VES generated significantly lower levels of gas, with hydrogen and carbon monoxide being most abundant.

The G-values are commonly used to estimate chemical yields of species generated by radiolysis and can be a useful means of comparing data. The G-value is usually defined by the amount of radiolysis product produced per 100 eV of radiation energy absorbed. As hydrogen was the
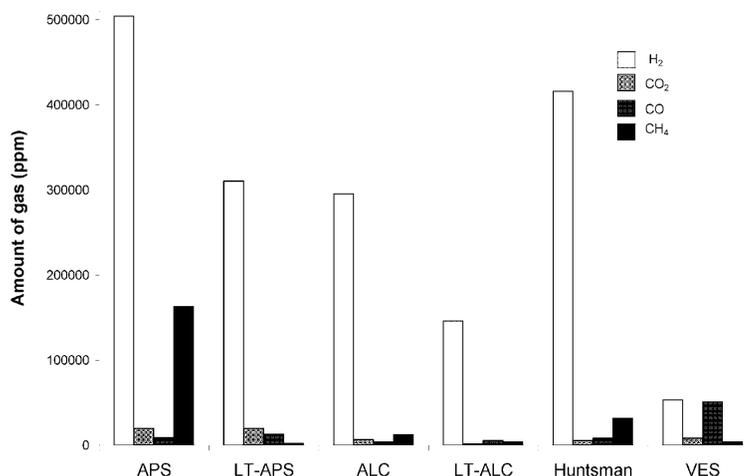

FIG. 4. Graph showing the amount of gases (ppm) generated by each of the candidate resins after irradiation to 10 MGy.

2991

most abundant gas for the irradiated encapsulants, $G(H_2)$ values have been calculated. Typical $G(H_2)$ values for APS, Alchemix, Huntsman and VES irradiated to 10 MGy were 0.43, 0.25, 0.33 and 0.04, respectively. Although data were limited for both LT-APS and LT-ALC formulations, the values of $G(H_2)$ were lower for equivalent ageing conditions when compared to the original formulations (0.27 and 0.13, respectively).

Leaching tests

The results of the TOC analysis (Table 4) show that in all cases except Huntsman, greater levels of TOC were leached at 80ºC compared to those aged at room temperature. This could, however, be due to increased diffusion of species such as additives out of the resins rather than any significant degradation. When irradiated, the concentration of organics in solution was lower than the corresponding, unirradiated solutions. This observation could suggest that, as the organic species were leached from the resin, they were subjected to radiolysis. This could have caused the organic species themselves to degrade down to lower molecular weight products which could have been lost in the gaseous phase as carbon dioxide for example. The TOC levels generated

TABLE 4. The TOC data (mg l^{-1}) for all epoxy resins and VES materials. The table compares TOC leached from both the thermally aged samples with those irradiated to 10 MGy at 4 kGy hr^{-1} (γ).

Material	Ageing conditions	Ageing time (days)						
		6	24	50	79	92	120	150
APS	pH 7 at RT	38	58	80	78	86	87	89
	pH 7 at RT & γ	–	21	3	7	3	4.5	5
	pH 7 at 80ºC	497	500	520	505	492	485	405
	pH 12 at RT	45	100	95	199	128	96	93
	pH 12 at RT & γ	–	18	5	7.5	6	7.2	14
	pH 12 at 80ºC	470	516	522	483	485	425	365
LT-APS	pH 12 at 80ºC	160	175	180	175	160	165	–
	pH 12 at RT & γ	4.3	14	2.6	2	3.2	6	–
Alchemix	pH 7 at RT	9	8	7.1	5.7	4.8	6.3	–
	pH 7 at RT & γ	7.1	3	3.3	1.2	2.2	1.9	–
	pH 7 at 80ºC	49	52	50	46	44	49	–
	pH 12 at RT	11	9	9.2	9.3	9.2	9.5	–
	pH 12 at RT & γ	8.1	3	4	2.3	3.7	3	–
	pH 12 at 80ºC	45	58	54	54	53	53	–
LT-ALC	pH 12 at 80ºC	11	15	18	22	–	–	–
	pH 12 at RT & γ	1.8	1.6	5	3	1.3	1.5	–
Huntsman	pH 7 at RT	2	2	4	3	4	6	4
	pH 7 at RT & γ	–	3.7	3.5	47	95	190	410
	pH 7 at 80ºC	27	38	45	73	55	51	58
	pH 12 at RT	44	71	59	140	106	7	5
	pH 12 at RT & γ	–	20	19	410	560	525	930
	pH 12 at 80ºC	63	102	95	49	79	51	53
VES**	pH 7 at RT	4	8	12	12	11	14	11
	pH 7 at 80ºC	126	128	134	107	112	97	95
	pH 12 at RT	36	83	83	167	114	44	40
	pH 12 at 80ºC	226	233	235	245	214	190	180

** No irradiation of VES in aqueous solution was carried out in this work because it has been reported previously by Poulson *et al*. (2006).

2992

by VES rose rapidly in the initial stages of ageing but reached a plateau before steadily decreasing. In the case of VES, there was a strong styrene odour noted when samples were removed and it is possible that some volatile organic components were lost from solution during sampling.

The concentrations of the organic compounds detected by GC-MS in the leachates from the APS, Alchemix and the low temperature compositions were generally low. However, the most commonly detected species was benzyl alcohol and in the unirradiated APS control solutions its concentration was as high as ~13000 µg l^{-1}. As benzyl alcohol was detected in the control leachates, it is suggested that its presence in solution is due to the fact that it is commonly used as a solvent/plasticizer in epoxy resin formulations, rather than as a degradation product. However, although the loss of benzyl alcohol might not be a consequence of the degradation of the epoxy resins, if it is used as a plasticizer, the loss of benzyl alcohol could result in a change in the mechanical properties of the resins. In the case of VES, the dominant leached organic species was dimethylaniline (DMA), which is also used during production of the resin.

The TOC levels detected in all the unirradiated Huntsman solutions peaked before then decreasing with increasing ageing time. The irradiated solutions, however, showed a significant increase over the ageing period; TOC levels of 930 mg l^{-1} were measured in the pH 12 solution after a dose of 10 MGy. The GC-MS did not detect any significant levels of organics in the Huntsman leachates, although the TOC levels at 10 MGy were very high. The reason for this observation is uncertain but it could be that the molecular weights of the organic degradation products were either above or below the detection threshold of the GC-MS analysis technique ($C_6 - C_{22}$).

Ion chromatography of the Huntsman solution irradiated to 10 MGy found significant levels of chlorides ($17-30$ mg l^{-1}) compared to the other irradiated epoxy resins. However, APS also generated significant chloride levels ($60-144$ mg l^{-1}) when thermally aged. The chloride could have arisen from residual chloride in the resins after conversion of epichlorohydrin, one of the building blocks used in the production of epoxy resins. The chloride levels leached from VES were negligible, perhaps as a result of differences in the manufacturing process as the vinyl ester component may be produced by esterification of an epoxy resin.

Conclusions

This study has reported the results of the exposure of six candidate epoxy resin based polymer materials: APS, LT-APS, Alchemix, LT-ALC, Huntsman epoxy resins and VES, to gamma irradiation. It also reports on the thermal ageing of unirradiated samples in aqueous environments of different pH. The aim of this series of experiments was to generate an empirical assessment of the radiation and thermal/chemical stability of the polymers.

The form of accelerated ageing described in this paper may not adequately account for environmental factors such as the ability of oxygen to diffuse through the test sample. In such cases diffusion-limited oxidation (DLO) caused by rapid oxygen consumption at high dose rates may give rise to dose-rate effects which can lead to an unreliably high estimate of radiation resistance. Work has recently started that supplements this test programme and studies the potential for dose-rate effects in APS and Alchemix epoxy resins.

The VES was the most stable of the candidate resins in terms of structural integrity. Previous studies by Poulson *et al.* (2006) have reported that the release of organic species from irradiated VES was low. Generally, all of the irradiated epoxy resin formulations showed similar changes in mechanical properties and leaching characteristics apart from Huntsman, which was so heavily degraded at 10 MGy that mechanical testing could not be carried out. The most abundant organic species detected in the epoxy leachates was benzyl alcohol, which is used during resin production, suggesting that it may not be a resin degradation product. The levels of chloride observed in solution could also be a consequence of the epoxy resin production process and may have implications for the corrosion of metallic components if present in high enough quantities.

The infrared spectra of the epoxy resins showed many similarities, both before and after irradiation to 10 MGy. Oxygen appeared to play a significant role in the degradation processes, as shown by the appearance of peaks associated with carbonyl species. A potential dose rate effect was observed using FTIR for the low temperature versions of APS and Alchemix.

The structural changes at 10 MGy associated with oxidation in the epoxy resins observed by FTIR, the reduction in gel fraction, the increase in solvent uptake ratio and the reduction in flexural

strength are suggested to be characteristic of chain scission in the resins.

Acknowledgements

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