Performance Test on Polymer Waste Form – 12137

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ABSTRACT

Polymer solidification was attempted to produce stable waste form for the boric acid concentrates and the dewatered spent resins. The polymer mixture was directly injected into the mold or drum which was packed with the boric acid concentrates and the dewatered spent resins, respectively. The waste form was produced by entirely curing the polymer mixture. A series of performance tests was conducted including compressive strength test, water immersion test, leach test, thermal stability test, irradiation stability test and biodegradation stability test for the polymer waste forms. From the results of the performance tests for the polymer waste forms, it is believed that the polymer waste form is very stable and can satisfy the acceptance criteria for permanent disposal. At present, performance tests with full scale polymer waste forms are being carried out in order to obtain qualification certificate by the regulatory institute in Korea.

INTRODUCTION

Boric acid wastewater and spent ion exchange resins are generated as a low- and medium-level radioactive wastes from pressurized light water reactors (PWRs). In Korea, boric acid wastewater is concentrated and dried in the form of granules (boric acid crystals), and finally solidified by using paraffin wax. Spent ion exchange resins are dewatered and stored in high integrity container (HIC). Recently, however, waste solidified using paraffin wax has been deemed suspect in meeting the acceptance criteria for permanent disposal. Also the use of HIC is limited due to its high cost.

In this study, polymer solidification was attempted to produce the stable waste form for the boric acid concentrates and the dewatered spent ion exchange resins. The polymer mixture which consists of epoxy resin, amine compounds and antimony trioxide was used to solidify the boric acid concentrates and the dewatered spent ion exchange resins.

To evaluate the stability of polymer waste forms, a series of standardized performance tests was conducted. Also, by the requirement of the regulatory institute in Korea, an additional test was performed to estimate fire resistance of the waste forms.

POLYMER

A polymer mixture consisted of two compounds, epoxy resin and polyamine hardener, was used to solidify the boric acid concentrates and the spent ion exchange resins. To improve the fire resistance, a small amount of antimony trioxide was added in the polymer mixture. The viscosity of epoxy resin is 12~22 poise and that of the hardener is 0.05~0.37 poise at temperature of 25 °C. The viscosity varies with the season. In winter, the viscosity of epoxy resin was 70~100 poise and that of hardener was 0.2~0.6 poise. Meanwhile, the viscosity of epoxy resin in summer was 2.1~7.1 poise and that of the hardener was 0.05~0.3 poise. The specific gravity of the epoxy resin was about 1.44 and that of the hardener was about 0.97 at temperature of 25 °C. The specific gravity of the epoxy resin also varies with the season. However, the difference of specific gravity with the season was not higher than that of viscosity. In winter, specific gravity of the epoxy resin was about 1.44 and that of the harden was 0.99 poise. In summer, specific gravity of the epoxy resin was about 1.43 and that of the hardener was 0.96 respectively.

It is recommended that the polymer mixture should be cured under the ambient condition of 15~30 °C. The Curing may be retarded at ambient temperature of less than 10 °C.

WASTE FORM

Lab Scale Waste Form

Polymer waste forms were prepared with the surrogate of boric acid concentrates and the surrogate of dewatered spent ion exchange resins, respectively. The surrogate of boric acid concentrates were quatzite granules of 2~5 mm in size. The surrogate of spent ion exchange resins was a fresh cationic ion exchange resins which is being widely used in nuclear power plants. First of all, the surrogate was poured into the cylindrical mold (length = 10 cm, diameter = 5 cm). After vibrating to compact the surrogate, polymer mixture was poured into the top of the mold. The poured polymer mixture flowed down through the voids of the surrogate and it was facillitated by sucking out the air inside the mold through a nipple attached on the bottom of the mold. The suction operaton was carried out by using vacuum pump. When the polymer mixture flowed out through the nipple at the bottom of the mold, which renders that the mold has been filled with polymer mixture, the suction operation was stopped and the compound of polymer mixture and surrogate was cured to a monolithic solid at room temperature for a period of time. After curing, the mold was removed and the waste form was subjected to the performance tests.

Full Scale Waste Form

The same materials as was used for lab scale waste forms were also used in preparation of full scale polymer waste form. Instead of the mold for lab scale waste form, a 55 gal drum and a 50 cubic feet drum were used to prepare the waste forms respectively. The full scale waste form was produced using the same procedure as for the prepation of lab scale waste form. After curing, a part of waste form was bored to get the specimens for the performance tests.

WASTE FORM PERFORMANCE

A series of performance tests required for the waste forms are summerized in Table I.

Compressive Strength

Compressive strength test was performed in accordance with ASTM C39 standard. The test results are shown in Table II for lab scale waste forms as cured, a mean compressive strength of the waste form containing surrogate of boric acid concentrates was 53.14 MPa (7,707 psi) and that of waste forms containing surrogate of dewatered spent ion exchange resins, 30.73 MPa (4,457 psi). For full scale waste forms as cured, a mean compressive strength of the specimens containing surrogate of boric acid concentrates was 39.70 MPa (5,758 psi) and that of the specimens containing surrogate of dewatered spent ion exchange resins, 26.97 MPa (3,912 psi). From the results of compressive strength test on waste forms, all the waste forms prepared for the tests were exhibited excellent structural integrity.

Thermal Stability

A thermal stability test was performed on the lab scale waste forms in accordance with ASTM B553 standard. By visual obsevation after testing, there was no significant change on the appearance of waste form. After thermal stability testing, compressive strengths of waste forms were measued and the results are shown in Table II. All the waste forms were turned out to have been retained their compressive strength after the testing.

Table I. Performance tests and methods on polymer waste forms

Test	Method
Compressive strength	- ASTM C39 [1], ASTM D5731 [2]
Thermal stability	- ASTM B553 [3]
Irradiation stability	- Exposure to total accumulated dose of 10^9 rad
Biodegradation stability	- ASTM G21[4] and ASTM G22[5]
Water immersion	- 90 day immersion in water - ASTM C39
Leaching	- ANS 16.1 [6]
Free water	- ANSI 55.1 [7]
Burning resistance	- KS M 3015(A) [8]

Table II. Compressive strengths of polymer waste forms

Waste form	Surrogate	As cured MPa (psi)	After thermal stability test MPa (psi)	After irradiation stability test MPa (psi)	After water immersion test MPa (psi)
Lab scale waste forms	boric acid concentrates	53.14±3.86[(a)] (7,707±560)	48.4±0.4[(c)] (7,015±55)	23.44±0.94[(d)] (3,400±136)	43.37±0.42[(e)] (6,290±61)
	spent resins	30.73±3.91[(a)] (4,457±567)	30.0±1.8[(c)] (4,346±256)	20.11±0.81[(d)] (2,917±117)	28.75±0.1.17[(e)] (4,170±170)
Full scale waste forms	boric acid concentrates	39.70±3.16[(a)] (5,758±459)	-	-	31.59±1.18[(f)] (4,582±171)
	spent resins	26.97±15.95[(b)] (3,912±2,313)	-	-	21.08±5.66[(g)] (3,057±821)

(a) Mean value and erroror at 95% confidence level for 10 replicates

(b) Mean value and erroror at 98% confidence level for 10 replicates

(c) Mean value and standard deviation for 3 replictes

(d) Mean value and erroror at 95% confidence level for 12 replicates

(e) Mean value and erroror at 95% confidence level for 3 replicates

(f) Mean value and erroror at 95% confidence level for 9 replicates

(g) Mean value and erroror at 98% confidence level for 9 replicates

Irradiation Stability

Irradiation stability test was performed on the lab scale waste forms under the exposure of total accumulated dose of 10^9 rad. Usually, irradiation stability testing on the waste forms is performed under the exposure of an accumulated dose of 10^8 rad. However, in this study, under more severe condition, the effect of irradiation on the polymer waste form was observed.

By visual observation after the test, there was no significant change on the appearance of waste form. No cracks or fractures were observed on the surface of waste froms. The compressive strength of irradiated waste forms were measued and the test results are shown in Table II. After irradiation, compressive strengths of the waste forms have exhibited a decrease of about 50% of the original strength in the case of waste form containing surrogate of boric acid concentrations, to about 75% in the case of waste form containing surrogate of spent ion exchange resins. However, because the compressive strength values of all the waste forms were more than 20.69 MPa (3,000 psi) after irradiation, it could be estimated that the waste forms still have enough structural integrity.

Biodegradation Stability

Biodegradation stability was performed on the waste forms in accordance with ASTM G21 standard(for determining the resistance of synthetic materials to fungi) and ASTM G22 standard(for determining the resistance of synthetic materials to bacteria). From the results of tests, no indication of culture growth was found on the polymer waste forms.

After a biodegradation stability test, compressive strengths of waste forms were measured in accordance with the ASTM D5731 because the waste form for the biodegradation stability test had a disc shape. The test results are shown in Table III. Polymer waste forms had still exhibited high compressive strength after the biodegradation stability test.

Table III. Compressive strengths before and after biodegradation stability test

Waste form	Surrogate	Before biodegradation stability test MPa (psi)	After biodegradation stability test	
			ASTM G21 MPa (psi)	ASTM G22 MPa (psi)
Lab scale waste forms	boric acid concentrates	46.76±1.21[(a)] (6,781±176)	39.30±0.62[(b)] (5,700±90)	40.34±0.35[(b)] (5,851±50)
	spent resins	29.07±0.54[(c)] (4,072±78)	23.32±0.57[(b)] (3,383±82)	24.47±0.84[(b)] (3,550±122)

(a) Mean value and standard deviation for 6 replictes

(b) Mean value and standard deviation for 9 replictes

(c) Mean value and standard deviation for 3 replictes

Water Immersion

For lab scale polymer waste forms, three replicates containing surrogate of boric acid concentrates and spent ion exchange resins respectively, were used for water immersion test. For full scale polymer waste forms, eight replicates containing surrogate of boric acid concentrates and spent ion exchange resins respectively, were used for water immersion test.

Each waste form was immersed in water at room temperature and was examined periodically. After 90 day water immersion, visual observation was made to find if any change and or defect of waste forms exist, and then a compressive strength test was performed on the waste forms in accordance with ASTM C39.

No change or defect on appearance were found in the waste forms during and after 90 day water immersion. The test results are summerized in Table II. Compressive strengths of the waste forms after tests were found to have been retained in the range of 80 ~ 90% of compressive strengths of the original waste forms before the test.

Leaching

Constant amounts of non radioactive cobalt, cesium and strontium were mixed with the surrogates. Then waste forms for the leach test were prepared with these surrogates. The leach test was performed in accordance with ANS 16.1 standard. The test resuts for the waste forms containing surrogate of boric acid concentrates and surrogate of spent ion exchange resins are shown in Table IV.

Leaching indices of cobalt and cesium could be measured from only one of three waste forms containing surrogate of boric acid concentrates. Leaching index of cobalt was 13.8 and that of cesium, 10.2. Strontium was not detected in the leachates.

For the other waste forms, cobalt, cesium and strontium in leachates were not detected during leach testing and the leaching index couldn’t be calculated.

Table IV. Resuts of leach test for the polymer waste forms

Surrogate	Waste Form	Element in lechate	Leaching index
Boric acid concentrates	# 1	Co	- These were not detected in the leahates.
		Cs	
		Sr	
	# 2	Co	13.8
		Cs	10.2
		Sr	- This was not detected in the leahates.
	# 3	Co	- These were not detected in the leahates.
		Cs	
		Sr	
Spent rein	# 1, # 2, # 3	Co	- These were not detected in the leahates for all the waste froms.
		Cs	
		Sr	

Free Standing Water

Free standing water test was performed on full scale waste forms in accordance with ANSI 55.1. The bottom of drum containing polymer waste form was punctured and then the amount of water flowing out from the bottom was measured. The drums with polymer waste forms containing surrogates of boric acid concentrates and spent ion exchange resins respectively, didn't have any free standing water.

Fire Resistance

Fire resistance test for the polymer waste forms was conducted in accordance with Korean standard, KS M 3015(A). From the resuts of the test, the polymer waste froms were found difficult to be fired and could be classified as fire retardant.

SUMMARY

Polymer waste forms were prepared with the surrogate of boric acid concentrates and the surrogate of spent ion exchange resins respectively. Waste forms were also made in lab scale and in full scale. Lab. scale waste forms were directly subjected to a series of the performance tests. In the case of full scale waste form, the test specimens for the performance test were taken from a part of waste form by coring.

A series of performance tests was conducted including compressive strength test, thermal stability test, irradiation stability test and biodegradation stability test, water immersion test, leach test, and free standing water for the polymer waste forms. In addition, a fire resistance test was performed on the waste forms by the requirement of the regulatory institute in Korea.

Every polymer waste forms containing the boric acid concentrates and the spent ion exchange resins had exhibited excellent structural integrity of more than 27.58 MPa (4,000 psi) of compressive strength. On thermal stability testing, biodegradation testing and water immersion testing, no degradation was observed in the waste forms. Also, by measuring the compressive strength after these tests, it was confirmed that the structural integrity was still retained. A leach test was performed by using non radioactive cobalt, cesium and strontium. The leaching of cobalt, cesium and strontium from the polymer waste forms was very low. Also, the polymer waste forms were found to possess adequate fire resistance.

From the results of the performance tests, it is believed that the polymer waste form is very stable and can satisfy the acceptance criteria for permanent disposal. At present, Performance tests with full scale polymer waste forms are on-going in order to obtain qualification certificate by the regulatory institute in Korea.

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