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Preliminarily Experiments of Liquid Scintillation Cocktail Waste Solidification

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Abstract

Liquid scintillation spectroscopy (LSC) is a widely used laboratory method for measuring radioactive especially low-energy alpha and beta emitter isotopes in solutions. Since the scintillation cocktails (SCs) contain large amounts of aromatic hydrocarbon compounds after the LSC measurement, the sample usually becomes organic radioactive waste, which is challenging to manage and dispose of. The most common and effective waste treatment method is incineration, a costly and cumbersome solution. Another simpler and less expensive way is the immobilization the organic liquids by embedding in cement or geopolymer matrix.

The aim of our experiments is to find a universal mix design to solidify and immobilize scintillation cocktail wastes (SCWs) of unknown composition. During the measurement, we assumed that the SCWs could contain Ultima Gold, Ultima Gold LLT, InstaGel Plus and ProSafe FC type scintillation cocktails in pure form or mixed with unknown quantities of water. Therefore, we performed experiments with inactive pure SCs as surrogates to mimic the SCWs. Since the water content determination of the organic solutions is difficult, the mix design must be independent of the water content of SCWs.

During our experiments, we found an appropriate mix design to solidify all available SCs up to 5% using CEM I 42.5 N type cement, Metaver N type metakaolin and sodium hydroxide. We could solidify our tested SCW with this mix, and this product meets the waste acceptance criteria of all Hungarian radioactive waste repositories.

Keywords

radioactive waste treatment, organic waste solidification, scintillation cocktail, Liquid Scintillation Spectroscopy, LSC waste

1 Introduction

Liquid scintillation spectroscopy (LSC) is a widely used laboratory method for measuring radioactive especially low-energy alpha and beta emitter isotopes in solutions. During the measurement the so-called scintillation cocktail is added into the sample, so the isotopes to be measured are mixed with the scintillator material. The radioactive particles directly excite the scintillator molecules, which emit photons in proportion to the radioactivity. Photons can be converted into an electrical signal using a photoelectron multiplier, which allows the radioactivity in the solution to be measured.

Scintillation cocktails (SCs) are organic solvents containing a large amount of aromatic hydrocarbon compounds. Thus, LSC analysis usually generates organic liquid radioactive waste (Fig. 1), which is difficult to manage and dispose of.

The most common and effective treatment method is the incineration of organic waste [1–3]; however, it is a costly and cumbersome procedure. Scintillation cocktails can also be theoretically regenerated by distillation [4], which can only be performed with sufficient airflow, and the quality of the regenerated SC is doubtful. Another way is to solidify liquid organic radioactive waste with special polymers [5, 6]. These materials are able to absorb multiple times of their weight, but they are mechanically vulnerable; and due to their consistency and leaching properties, it is necessary to provide secondary packaging. Embedding this type of waste in a cementitious or geopolymer matrix may be a safer and less costly treatment option for long-term storage [7–17].

The international radioactive waste management practice aims to reduce the amount of radioactive waste generated in accordance with the recommendations of the Inter-

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Fig. 1 Storage of liquid scintillation cocktail waste

national Atomic Energy Agency (IAEA); and instead of liquid waste, produce solidified waste packages with sufficiently low dissolution properties [18–20] and safe storage.

There are currently two radioactive waste repositories in operation in Hungary: the National Radioactive Waste Repository (NRWR) in Bátaapáti and the Radioactive Waste Treatment and Disposal Facility (RWTDF) in Püspökszilágy. NRWR prescribed two essential requirements on cemented waste from the Paks Nuclear Power Plant: the compressive strength of the hardened cement paste must be at least 10 MPa at 28 days; the diffusion coefficient for each isotope must not exceed 10^{-7} cm^2/s [21]. Radioactive organic waste of non-nuclear origin, i.e., from other institutions should be deposited at RWTDF, and the organic waste must be adsorbed. According to the waste acceptance criteria, the volume of the initial organic solvent should not exceed 10 V/V% of the final volume; in contrast, the total weight of the solution and sorbent should not exceed 25 w% of the total mass. The compressive strength of the cemented product must be at least 0.3 MPa, and the diffusion coefficient must not exceed 5×10^{-7} cm^2/s for tritium and 3×10^{-9} cm^2/s for cesium [22].

Compressive strength is closely related to porosity and permeability [23, 24], which significantly determines the leaching properties of hardened cement paste [10, 25]. Thus, the compliance of compressive strength and diffusion coefficient values is important for the long-term safe storage of solidified waste. To this end, the compressive strength of the specimens containing SC and SCW (Scintillation Cocktail Waste) was determined according to EN 196-1 [26] and the diffusion coefficient of ^{3}H according to ASTM C1308-08 [27] standards.

2 Experimental

The primary purpose of our experiments is to develop a universal mix design, which can be used to convert various types of scintillation cocktail waste (SCW) of unknown composition into solid form, at low cost, under laboratory and pilot conditions. It can avoid the costly and cumbersome transportation of liquid organic radioactive waste e.g., to a foreign country for incineration.

In this study, we investigated the cementation of Ultima Gold, Ultima Gold LLT, Insta-Gel Plus, and ProSafe FC scintillation cocktails (SCs) as surrogates of SCWs using cement and geopolymer-like matrices and compliance of the solidified products with the waste acceptance criteria.

The main components of each cocktail are shown in Tables 1 and 2 [28–31].

Using the experimentally determined appropriate mix design we solidified one of the stored SCWs.

It has been assumed in the experiments that the waste consisted of substances listed in Tables 1 and 2, in pure form or mixed, with an unknown water content. According

Table 1 The main composition of scintillation cocktails [28, 29]

Component	Ultima Gold	Ultima Gold LLT
Diisopropyl naphthalene isomers	60–80%	40–60%
Alkylphenol Polyglycolether	10–20%	20–40%
Diisooctyl acid phosphate	2.5–10%	-
Phosphoric acid, 2-ethylhexyl ester	2.5–10%	-
2-(2-butoxyethoxy) ethanol	-	10–20%

Table 2 The main composition of scintillation cocktails [30, 31]

Component	Insta-Gel Plus	ProSafe FC
Diisopropyl naphthalene isomers	-	50-80%
Alkylphenol Polyglycolether	40–60%	
2-(2-butoxyethoxy) ethanol	-	5–10%
Alcohols, secondary C11-15, ethoxylated	-	20–40%
1, 2, 4-trimetilbenzene	40–60%	-

to PerkinElmer's recommendations, a maximum of 10 cm^3 sample can be tested by 10 cm^3 of Ultima Gold during the LSC measurement; in case of Ultima Gold LLT > 10 cm^3, and Insta-Gel Plus may contain 10 cm^3 of the sample to be tested by LSC, depending on the quality and concentration of the target sample [32]. Regarding the deionized water content, a maximum of 3.2 cm^3 is recommended for Ultima Gold and only 1.7 cm^3 for Insta-Gel Plus per 10 cm^3. Theoretically, there is no such restriction (> 10 cm^3) for Ultima Gold LLT [32]. No similar data were found for the ProSafe FC cocktail, but in our experience, 1.7 cm^3 of deionized water started the gel-formation in the mixture.

Initially, we tried to find a simple mix design that could solidify any possible scintillation cocktail regardless of its water content in an economical way. Our pilot test experiment was performed by the direct method of [17], using inactive SCs as a surrogate and CEM I 42.5 N cement with various w/c ratios (0.25–0.40). The pilot mixings were performed on a small scale (ca. 80 cm^3), and the properties of the cement paste (bleeding, cracking, texture) were observed visually and using a Vicat apparatus. During these experiments, first we tried to solidify Ultima Gold LLT type scintillation cocktail using only CEM I 42.5 N cement with a variable scintillation cocktail content of 5–30% and with a variable w/c ratio of 0.25–0.40 (Table 3).

Since each of these samples were hardened, we tried to adopt this mix design to Ultima Gold type scintillation cocktail, but we could only achieve a maximum of 5% dosage of Ultima Gold (Fig. 2, bottom left).

Dianu and Podină [7] used Portland cement, aluminum stearate ($C_{54}H_{105}AlO_6$), lime, and sodium silicate to solidify Ultima Gold XR scintillation cocktail. With this method, less than 1.16×10^{-8} cm^2/s (10^{-3} cm^2/day) diffusion coefficient was achieved in the case of ^3H; on the other hand, compressive strength of 5×10^{-2} MPa (50×10^3 N/m^2) was measured. Although in our case these values do not meet the NRWR waste acceptance requirements, we tested the aluminum stearate Dianu and Podină [7] considered to be the best in several concentrations; however, we could not further increase the content of the Ultima Gold in the cement paste.

For the second part of our experiments we used the same type of ordinary Portland cement (CEM I 42.5 N) as for the pilot test substituted with different amounts of metakaolin. The experiments were performed with 10–50% Keramost KM60 or Newchem Metaver N type metakaolin substitutions. The composition of the metakaolins used is shown in Table 4. With this method, all SC tested can be solidified at a 5–30% related to the amount of cement with a little bleeding.

Ultima Gold can be solidified up to 15% with metakaolin type KM60, but it can be solidified up to 30% using Metaver N with the presence of 25 w% NaOH. This mix proportion can be used in the case of each SC tested. The best results were obtained using 43% Metaver N and 25 w% NaOH solution, the exact mix design is shown in Table 5. In this table one can see ranges of SC and deionized water; these values depend on the assumed water content of the SC.

Table 3 Solidification mix design for Ultima Gold LLT

Constituents	Mass [g]
Cement (CEM I 42.5 N)	100
Deionized water	25–40
Ultima Gold LLT	5–30

Fig. 2 The stages of the mix design development

Table 4 Composition of metakaolins used

Component [%]	KM60	Metaver N
SiO_2	50–55	52–53
Al_2O_3	min. 40	43–44
Fe_2O_3	max. 1.45	< 1
CaO	0.05–0.5	< 0.5
MgO	0.20–0.45	< 0,4
$K_2O + Na_2O$	max. 1.5	< 1

Table 5 Composition of the developed mix design

Constituents	Mass [g]
Ultima Gold	0–150
Metaver N	150
Cement (CEM I 42.5 N)	350
NaOH 25 w%	50
Deionized water	150–225

With these constituents, any SCW with up to 50% water content can be solidified; however, dissolved salts of unknown composition can affect the cement setting.

We shall mention that the SC content has a plasticizing and set retarding effect on cement pastes [8–10, 17, 24].

Fig. 2 shows the stages of the mix design development. It can be seen that using only cement and 30% of Ultima Gold the mixture cannot be solidified (top left) and remains in gel phase, but using metakaolin 10% KM60 the mix can be solidified in the form of granulate (top right). The bottom left image shows the texture of the cement paste using only cement with 5% of Ultima Gold content. Finally, in the bottom right picture one can see the final mix design using metakaolin Metaver N and NaOH (Table 5).

The radioactive SCW can also be solidified with this mix proportion up to 30% SC content. However, the SC began to dissolve within a few hours from the specimen of Ø25 × 50 mm prepared according to the ASTM C1308-08 standard [27]. After 24 hours, SC appeared on top of the leachate (Fig. 3) in a separate liquid phase.

It should be noted that with the mix design shown in Table 6 (CEM I 42.5 N type cement, 43% Metaver N and 25 w% NaOH solution) each of the SCs can be solidified. Still, this product was not acceptable without secondary packaging due to its poor leaching properties.

Since the result of the mix design cannot comply with the waste acceptance criteria of the NRWR, we had to modify the procedure further. Therefore, we tried to follow the studies of [8, 11] where Monophase 40 scintillation cocktail was cemented using Arkopal N-100 (nonylphenol polyglycol ether) surfactant. Bayoumi [8] found that the optimal w/c ratio was 0.35 to achieve acceptable workability and compressive strength. According to his measurements, the optimum SC concentration of the cement paste was found to be 10% with the addition of 0.15 w% surfactant, which gave a compressive strength of 14.2 MPa, but the leaching property of ^{14}C significantly deteriorated upon solidification of 15% SC. In our case, the recommended w/c ratio [8] of 0.35 did not allow achieving proper workability of the cement paste, certainly due to the water uptake of Metaver N type metakaolin. However, it became clear that in order to achieve a better leaching property, the SC content of the mixture had to be reduced.

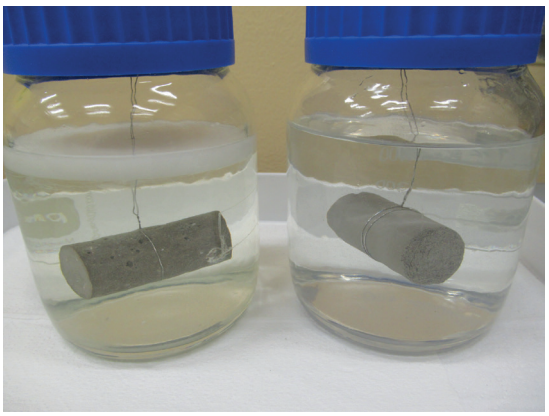

Fig. 3 Leaching test: The appearance of the new organic liquid phase (left) and the appropriate specimen

Based on the study of El-Naggar et al. [14], we first added SC and SCW to metakaolin, then mixed with deionized water. Then CEM I 42.5 N type cement was added to prepare a homogeneous cement paste. After thorough mixing, a solution of 25 w% NaOH was finally added to give an easy-to-apply geopolymer-like material (Fig. 4). There was no need to use additional surfactants because the SCs already contain them [33–36]. The exact composition of this mix design is shown in the Table 6; the ranges of the SC and deionized water depends on the hypothetical water content of the SCWs. Example: In the case of 2.5% SCW with 20% water content of the solidified product 12.5 g SCW may contain 10 g SC and 2.5 (20%) water but the amount of additional deionized water is constant 150 g.

During the experiments, 40 × 40 × 160 mm sized specimens were prepared using SC with an additional deionized water content of 0, 10, 20%. The w/b ratio was kept at 0.6 to achieve good workability and proper homogeneity. The setting time of the composite paste was found between 50 and 60 minutes, while the final setting time was within two hours from the cement being added to the mix. In our experience, up to 10% SCW can be solidified with this method because at higher concentrations, bleeding occurs.

After demolding, the specimens were placed in a sealed bag and stored in a climate chamber at 20 °C and 90% RH. The compressive strength was measured at the age of 28 days, according to the EN 196-1 [26] standard, with a load rate of 2400 ± 200 N/s, on two half-prisms, so the results in Table 7 are the averages of two compressive strength values.

Table 6 Composition of developed mix design

Constituents	Mass [g]
Scintillation cocktail	10–25
Metaver N	150
Cement (CEM I 42.5 N)	350
NaOH 25 w%	200
Deionized water	150–155

Table 7 Compressive strength of hardened cement pastes made using various SCs with different water contents

Scintillation cocktail	Water content of SC [w%]	Compressive strength [MPa]	
		2.5% SC content	5.0% SC content
Ultima Gold	0	15.71	12.44
	10	14.63	13.53
	20	13.59	14.27
Ultima Gold LLT	0	14.53	17.07
	10	14.45	16.28
	20	14.58	14.62
Insta-Gel Plus	0	16.29	14.84
	10	16.34	15.56
	20	16.63	16.90
ProSafe FC+	0	16.10	14.47
	10	16.67	14.24
	20	16.92	14.02

The Fig. 4 shows the steps of the mixing and the specimen preparation process. First the 25 w% NaOH was diluted with deionized water in order to utilize the heat of hydration later. Then 150 g Metaver N type metakaolin was mixed with 10.0–12.5 g (2.5%) or 20.0–25.0 g (5.0%) SC depending on its hypothetical water content (top left). Thereafter 150 g deionized water and 350 g CEM I 42.5 N type cement were added to this mixture (top right). Finally, 200 g 25 w% NaOH at 50 ± 5 °C was added (bottom left) to the mix, then standard specimens were prepared for compressive strength and diffusion coefficient measurements.

As expected, the compressive strength results showed that the values of each sample decreased with increasing organic matter (SC) content (Table 7). We observed an increase in compressive strength with the increasing SC content only in the case of the Ultima Gold LLT, but as the water content increases, this improvement ceases.

In general, the water content of the SC has only a little effect on the compressive strength. However, contrary to the preliminary expectations, the compressive strength of the 2.5% Ultima Gold and 5% Ultima Gold LLT decreases with increasing water content in the SC.

The effect of the organic matter content on the compressive strength can also be seen in the case of SCW. It must be noted a blank sample also was prepared without SC, using only deionized water and its compressive strength was 16.36 MPa. It means that the mix design can be applied regardless of the water content of the SCWs, moreover decreasing of the SC content increases the compressive strength of the cemented product.

Table 8 shows the compressive strength of the mixtures decreases with increasing SCW content, and above 5%, no measurable results were obtained according to EN 196-1. In the case of the samples containing 7.5 w% and 10 w% SCW, the half prisms were not cracked at an exact load but collapsed. We performed the leaching tests on specimens of 25 × 50 mm prepared with SCW according to the ASTM C1308-08 [27] standard at 26 ± 1 °C with drinking water.

The cylindrical specimens were immersed in drinking water (Fig. 3) because of its low tritium concentration, and it was replaced with fresh water at intervals specified in the standard. Then the ^{3}H and ^{60}Co, ^{134}Cs and ^{137}Cs concentrations of each leachate were measured, from which the diffusion coefficients could be calculated using Fick's laws. The composition of the solidified SCW is shown in Table 9.

The radioanalytical measurements showed that the activity concentration of ^{60}Co and ^{134}Cs ^{137}Cs isotopes was below the limit of detection. The samples of the LSC measurement

Fig. 4 The process of specimen preparation

Table 8 Compressive strength of cement pastes made using SCW at various waste contents

SCW content [w%]	Compressive strength [MPa]
0.0	16.39
2.5	15.44
5.0	12.97
7.5	non-measurable
10.0	non-measurable

Table 9 Radiochemical composition of the cemented SCW

Isotope	A [Bq/dm^3]
^{60}Co	4.93
^{134}Cs	9.89
^{137}Cs	771
^{3}H	1.44E05

of the leachate also contained up to 24 Bq/dm^3 ^{3}H concentration; thus, the diffusion coefficient of each nuclide could not be determined.

3 Conclusions

As a result of our experiments, we established the following:

I. Ultima Gold LLT type scintillation cocktail can be solidified using only CEM I 42.5 type cement even at 40% (related to the amount of cement) cocktail content, but we achieved only 5% with the Ultima Gold type scintillation cocktail.

II. Ultima Gold, Ultima Gold LLT, ProSafe HC+, Insta-Gel Plus scintillation cocktails and the liquid scintillation cocktail waste (SCW) with unknown composition can be solidified using the following mix design and order:
   1. 150 g Metaver N type metakaolin
   2. up to 25 g scintillation cocktail waste
   3. 150 g deionized water
   4. 350 g CEM I 42.5 N type cement
   5. 200 g 25 w% NaOH at 50 ± 5 °C.

All measured SC and an SCW can be solidified with this final mix design up to 5%. Regarding the leaching properties of each isotope, it was found that the diffusion coefficients of the solidified SCWs as described above according to ASTM C1308-08 [27] met the waste acceptance criteria for both Hungarian radioactive waste repositories. Based on these results, it can be concluded that the solidified products meet the NRWR acceptance criteria, as their compressive strength exceeds 10 MPa according to EN 196-1 [26], and the diffusion coefficient of each isotope is less than 10^{-7} cm^2/s, respectively.

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