

SOLIDIFICATION AND STABILIZATION OF OIL DRILL CUTTING MUD WASTE USING PORTLAND CEMENT(PC) AND POROTHERM RED BRICKS POWDER (PRB) WASTE

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ABSTRACT

In this study, solidification/stabilization (S/S) of heavy metals (HMs) (lead, (Pb); copper, (Cu); zinc, (Zn); nickel, (Ni); and chromium, (Cr)) using mixture of Portland cement (PC) 15% and Porotherm red bricks (PRB) powder waste with a ratio of (5, 10 and 15%) resultant (3:1, 3:2 and 3:3) cement/Porotherm solid monolith and degraded ones with three replicates was carried out. Physical and chemical characteristics were investigated. The physical characteristics studied were bulk density and unconfined compressive strength (UCS) while chemical characteristics studied were leachability of those aforementioned HMs using Toxicity Characteristics Leaching Procedure (TCLP) and the change in leachate pH. Results indicated the optimum mix design for the S/S system of HMs drilling fluid using both PC and PRB at the experimental conditions in terms of reuse of PRB waste in s/s. However, the study showed that three levels of mixes studied (5%, 10%, and 15%) were able to immobilize the HMs at very low concentration far beyond the regulatory limits by (US EPA). The UCS at above mentioned concentration was 0.811, 4.21 5.1 Mpa respectively and PRB waste powder was effective to reduce HMs leachability and increase compressive strength.

Keywords: Solidification, Stabilization, Mud Waste, Portland Cement, Porotherm Red Bricks

1-INTRODUCTION

Petroleum companies generate tones of oily sludge of tanks bottom, oil contaminated soil and drilling mud waste. Yet, those contaminants of mud cuttings usually travel to neighboring environments by the action of wind or/and water and consequently to the underground water through the soil profile by spontaneous infiltration or the action of rain. Solidification and stabilization of the drill cutting will, preventing the environment from the toxic contaminants. Drill cuttings are soil-like wastes, which containing a significant hazardous materials, hydrocarbons, salts, heavy metals [1]. Physical and chemical properties of drill cuttings significantly affected by several factors such as type of field

geology, drilling fluid used (water-based, oil-based or synthetic fluids), oil well location, drilling techniques, rig operator [2, 3]. The solidification is the process which converts the waste to available storage, landfill or reuse materials, and stabilization is a chemical process to minimize and reduce the contaminants hazardous [3]. Many binder materials used for stabilization and solidification such as Portland cement, fly ash, plaster, lime, zeolites and other compounds but cement and or fly ash is the most viable to solidification and stabilization [4] lime, bentonite, fly ash, clinker and gypsum were used for landfilling and roads construction application [5]. Chelating agent was more effective than inorganic agent's (sodium, sulfide and lime [6]. The pH changes, affect heavy metals leaching [7] nitrate solution enhance stabilization and solidification of Pb(II) and Cr(III) [8] using Portland cement (CEM I), with the addition of high carbon power plant fly ash (HCFA), effect positively the unconfined compressive strength (UCS), hydraulic conductivity, porosity, leachate pH, and acid neutralization capacity (ANC) [9]. Industrial byproduct, anhydrite (CaSO_4) can significantly reduce the leached metal concentrations [10]; the compressive strength was 2.52–12.7 MPa for 60% cement mixed with 40% fly ash and 6.62–16.12 MPa for a mixture of 60% cement and 40% bottom ash. The compressive strength reduced to 0.55–1.30 MPa when 30% cement was mixed with 70% fly ash and to 0.90–7.95 MPa when 30% cement was mixed with 70% bottom ash, respectively [11]. Cement solidification/stabilization technology has significant effect in immobilizing Cr and Ni [12]. A fly ash reduced (As) and lead (Pb) by 98.3% and Pb release was reduced by 98.5%. from field soil samples contaminated with arsenic [13]. Leaching metals was strongly pH dependent [14]. Matrixes with heavy metals-bearing sludge and 5–15% fly ash with ordinary Portland cement (OPC) was sufficient to achieve the target compressive strength of 0.3 MPa required for landfill disposal [15]. The 15 wt. % oil palm ash OPA, 35 wt.% ordinary Portland cement OPC and 50 wt.% Ni contaminated sludge increase nickel hydroxide stabilization [16]. Such processes raise up the advantage of materials locally available from Porotherm Red Bricks waste resulted from Porotherm Red Bricks (PRB) industry for S/S technology and to implement the S/S technology for HMs remediation.

2 MATERIALS AND METHODS

2-1- Drill cutting waste

Soil (drill cutting mud) which was collected from an unidentified Libyan oil production field sited at Ghdamas city territories, at 400 km south-west of the capital Tripoli, The binders are Porotherm Red Bricks waste from Porotherm Red Bricks plant 60km south Tripoli and the OPC from cement industry plant. Physical and chemical properties were illustrated in table 1, 2 and 3 respectively.

Table 1:
physical and Chemical properties of the waste drilling mud.

Moisture %	mg/kg					
	THC**	Pb**	Cu**	Zn**	Ni**	Total Cr***
28.29	55.8	7.4	7.4	63.6	5.6	9.9

***EPA 5035A; 2002 + EPA 8015D;

* THC: Total Hydrocarbons 2003

****EPA 3150B; 1996 + EPA 6010C;
2007

Table 2:
physical and Chemical properties of the PRB waste.

KAlSi3O8	Fe2O3	SiO2	Ca2SO4	Bulk density	PH
20.43%	12.26%	51.99%	15.32%	1.88 gm/cm ³	12

Table 3:
Some physical characteristics of PC

Expansion/1.70 mm		> 10 mm
Time of	Initial (1.0 h + 45 min)	> 45 min
	Terminal (>	
Solidification	3.0 h)	< 10 h
	3 days, 28.8	
Compressibility	N/g2	> 21 N/g2
	28 days, 49.5	
Strength resistance	N/g2	> 39 N/g2

*Libyan standard analysis values. 340/1997.

2-2- EXPERIMENTAL FACTORS

In this part of the study, two factors were examined for their effect on the final S/S treatment product and they were as follows:

2-2-1- BINDER-TO-SOLIDIFIER-TO-WASTE RATIO STUDY

Experimental runs were carried out at three levels of drilling mud, OPC and PRB powder mixes (5%, 10%, and 15%) while keeping the OPC at constant (15%) to assess the effect of the PRB binder on the final S/S product.

2-2-2- Curing Time

Tests were carried out on the S/S final product at different times: 24 hr., 3 days, 6 days, 12 days, 18 days, 23 days and 28 days.

2-3 Testing Procedure

A set of physical and chemical experimental tests were carried out on the different mixes of S/S final products. To evaluate the degree of fulfillment of the S/S system objectives specific criteria [17]

2-3- METHODOLOGY

2-3-1-UNCONFINED COMPRESSIVE STRENGTH

Unconfined Compressive Strength (UCS) test was performed to study the progress of hydration reactions. The test carried in a conventional method according to the test method. The procedure commences with crushing the stabilized sample and allowing it to pass through a 20.00 mm test sieve. A UCS cubic mold of 10.00 cm was filled with a calculated mass of the stabilized material. This was compacted using a hammer to drive home the upper plunger. The sample was ejected with an ejecting plunger and weighed to the nearest 1.00 g. The sample was wrapped with a cardboard paper, waxed and stored at a temperature of 20°C for six days, after which both were removed and the sample weighed to the nearest 1.00 g. Then, the sample was placed in a water bath for 24 h, removed and allowed to drain for 15 min. The compression testing machine plunger was set under a CBR ring capacity of 50 kN and the sample crushed at a uniform rate of 1.00 mm/min. Readings of the maximum force required to shear the sample were recorded [17]

2-3-2- LEACHABILITY ANALYSIS (TOXICITY CHARACTERISTICS LEACHING PROCEDURE, TCLP)

TCLP tests were performed according to toxicity characteristics leaching procedure (TCLP) US- [18] test method to evaluate the adsorption behavior (and the effectiveness of immobilization treatments) of the studies HMs (Pb, Cu, Zn, Ni, and Cr) in the matrices of different ratios of (Binder : Solidifier : Drilling mud) for different curing times (1, 3, 6, 9, 15, 20 , and 25 days) using samples made of drilling mud, PRB binder, and PC as a solidifier. Weighed quantities of those aforementioned materials and water were homogenized in a mixer. The freshly prepared mixtures {(5%, 15%, 80.0%), (10%, 15%,

75%) and (15%,15%,70%)} respectively was poured into 10.0 cm x 10.0 cm x 10.0 cm steel cub mold separately and to be filled to the top of it. The full in-filled mould was compacted using vibrating table for approximately 30 sec and then again filled completely. The mould was re-compacted and the excess of the mixture filled in a gain is scraped off to obtain a flat and smooth surface. The mold were then sealed in plastic bags to prevent possible carbonation due to exposure to air and cured for 24 h in a humidity chamber with a relative humidity of 98% and a temperature of 24 °C before de-molding. The re-mold sample is resealed once again in plastic bags and transferred back into the humidity chamber for further curing prior to testing.

2-3-3- Analysis of Leachate Concentrations of HMs by Inductively Coupled Plasma – Joined with Optical Emission Spectroscopy (ICP-OES).

Measurements of studied metals (Pb, Cu, Zn, Ni, and Cr) was carried out using Inductive Coupled Plasma – Optical Emission Spectroscopy (ICP-OES), For each item, three measurements were carried out and arithmetic mean value was calculated with standard deviations. Besides, the waste samples had water content of $28.29\% \pm 1.53$ and THC of 50.6 ppm.

3- RESULTS AND DISCUSSION

Following to the chemical characterization of the drilling mud shown in table 1, the main contaminants were found to be Pb, Cu, Zn, Ni, and Cr with harmful amounts which affect fauna and flora.

The main chemical compositions of PC is CaO, 64.04%; SiO₂, 21.28%; Al₂O₃, 5.85%, Fe₂O₃, 3.31% and SO₃, 2.30 which are considered as the most abundant and typical constituents of PC in compliance with the standards [19]. The PC reduces the mobility of the inorganic compounds by formation of insoluble hydroxides, carbonates, or silicates; and substitution of the metal into a mineral structure. The presence of Calcium oxide (CaO) in high percentage is the most essential substance in S/S treatment due to its strength building characteristics. Whereas, silicates (SiO₂) and aluminum oxide (Al₂O₃) are two main components responsible for the formation of CSH (calcium silicate hydrate) and Etringite hydrate respectively where the HMs are most stabilized in the matrix [15]. PRB powder binders are usually combined with PC stabilizer to form an admixture to enhance or add desirable properties to concrete such as strength, reduce leach-ability of contaminants, easy of handling, waterproofing and durability properties and decontaminate the industrial waste PRB.

The composition of PRB waste powder as table 2 indicated high percentage of CaSO₄ as compared to the PC. It can be postulated that CaSO₄, once reacted with the leachant water, may produce acidic conditions and lead to pH shift towards neutrality.

The moisture content is determined as the mass of free water that can be removed from a material, usually by heating at 105°C, expressed as a percentage of the dry mass [20] If the amount of moisture present in a binder can affect strength in the S/S product. Low percentage of water content (0.39 %) has

resulted definitely in less porosity and permeability, and a lower bulk density (1.96% as wet mass; 1.88% as dry mass) of the final compacted product all of which are favorable in S/S system. The particle size distribution of the binder material is playing a key role in s/s technology.

Table 4:

Mean values of bulk density and compressive strength of the mixes PC and PRB

Cupic mix %	Bulk density gm/ Cm ³	compressive strength (Mpa)
5	1.4	0.811
10	1.42	4.21
15	1.53	5.1

The results in table 4 shows that each of the three levels of mix; 5.0%, 10.0%, and 15.0% had an average bulk density of 1.40 g/ml, 1.42 g/ml, and 1.53 g/ml respectively. These values demonstrate homogeneity in settings among the three mixes were almost free of internal air voids.

3-1 Compressive Strength of (5%, 10%, 15%) Levels of Mixes

It has been reported that the high UCS is not necessary for most waste management scenarios, however, can be so when utilized for instance in production of construction materials [21]. Landfills disposals of solid wastes, is recommended to conform minimal compressive strength requirements in Resource Conservation & Recovery Act's; that is 0.3 MPa [15]. The test is known as a good indicator for the progress of the hydration reaction in cement S/S systems. Table 4 shows the obtained results from S/S system at the end of curing time (28 days). The 5% level of mix (the highest mix in waste drilling mud, (80%)) showed an intensive decrease in compressive strength of 0.811 MPa as compared to the 15% level of mix which had a compressive strength of 5.102 MPa at the end of curing time of 28 days. This could be attributed to a chemical reason; the HMs oxides (Pb, Cu, Zn, and Cr) present in the drilling mud can cause retardation of cement paste and stop production of main cement hydration products such as calcium silicate hydrate (C-S-H) and pontlandite (Ca(OH)₂) crystals which is known to withstand the mechanical forces. [22]. However, others attributed to the presence of a physical reason [23]. This was represented by the presence of fine particles of soil (the drilling mud) with high surface areas, which might effectively reduce the amount of cement available for binding the fine and coarse aggregates required. On the other hand, reducing the surface tension will directly affecting the adhesion related to the phases (cement, aggregates) [24].

3-2 Leachability Analysis (TCLP Test)

According to US EPA TCLP standards, the limit of TCLP leaching concentration of Pb, Zn, Ni, and Cr are 5.0, 300, 7.0 and 5.0 mg/l respectively.

Interaction of HMs contaminants with waste soil (drilling mud) and/or binder involves a number of fixation mechanisms such as: (i) adsorption to binder-soil matrices which depends in soil mineralogy, (ii) pH-dependent precipitation where salts are formed and precipitated from solution to facilitated the surface adsorption of HMs ions; (iii) adsorption/encapsulation into and onto nano-porous C-S-H gel which confers a high specific surface area (between 10 and 50 m²g⁻¹) and (v) incorporation into crystalline components of the cement matrix.

Table 5:

pH measurements during the curing time of the TCLP test in respect to the cubic samples

Curing time (day)	1	3	6	9	12	19	24	30
pH at (5% level)	10.1	9.19	10.93	10.96	10.85	10.85	10.80	10.65
pH at (10% level)	8.30	8.00	8.90	8.80	8.90	9.10	9.50	9.50
pH at (15% level)	9.50	8.25	9.75	9.50	10.00	10.00	10.00	9.75

Table 6:

pH measurements during the curing time of the TCLP test in respect to the grounded samples

Curing time (day)	1	3	6	9	12	19	24	30
pH at (5.0% level)	10.3	8.55	8.40	8.30	8.20	8.10	7.90	7.90
pH at (10.0% level)	8.00	7.85	8.86	8.70	8.20	8.20	8.05	8.05
pH at (15.0% level)	8.30	7.05	8.50	8.33	8.15	8.10	8.10	8.09

TCLP tests for the compacted cubic mold and their grounded samples were run in distilled water initially at neutral pH of 7.0. However, leachants in cubic mold samples had an increase in their pH ranging between 8.5 – 10.5 during the course of curing time (1 - 25 days), with most over 10.75 for the 5% level of mix, and 9.0 for the 10% and 15 level of mixes (table 5). In contrast, the ground S/S samples had pH value lower than (7.5 – 8.5),(table 6). It is documented that for S/S systems with pH value (7.0 -- 11.0) is usually recommended. However, a higher value might require reducing the mass percentage of cement in the binder [25]. Regarding to the cubic mold samples (table 5) showed an increase in pH

from initial original pH 7.0 to pH 10.50. Overall, this result can be attributed to the gradual formation of cement hydration product (C-S-H) coexisting with (CaOH), the most alkaline hydration product in cement-based system [26]. It seemed that the increase of PRB mass percentage from 5.0% to 10.0% and 15.0% had attenuated the action of PC hydration and thus the production of CaOH responsible for pH increase.

From table 6, it can be observed that the grinding of S/S final products, has decreasing the leachant pH value in the S/S. The retaining of pH of the ground sample leachant at neutrality is attributed to Physio chemical changes due to grinding of monoliths and the presence of amphoteric HMs contaminants such as Pb and Ni [27]

Table 7.**Leachability concentration before and after the TCLP test**

Element	Initial value (before TCLP test) \pm SD (ppm)	Final value (after TCLP test) \pm SD (ppm)*	Leachability by mix level, (%)		
			5.0	10.0	15.0
Pb	7.4 \pm 1.2	1.2 \pm 0.9	99.8	83.8	97.0
Cu	7.4 \pm 2.2	0.025 \pm 0.002	98.1	99.7	99.7
Zn	63.6 \pm 10.2	0.012 \pm 0.015	98.3	\approx 100	\approx 100
Ni	5.6 \pm 0.9	0.148 \pm 0.08	95.7	97.5	96.7
Total Cr	9.9 \pm 2.1	0.003 \pm 0.0004	97.2	\approx 100	\approx 100

* Final values chosen by best level of mixes; that is the 10% level

The leachant concentration of the studied metals (Pb, Cu, Zn, Ni, and Cr) in a 10% level of mixes at the end of curing time (25 day) were considerably lower than the initial concentration before the TCLP test (table 7). The results revealed that the Pb has a less decrease in final leaching concentrations 10% PRB and other elements decrease with PRB% increase:

Extending the curing time (aging) means more hydration reaction will take place and as a result, more metals will be stabilized in different chemical forms, which will present in either the developing crystalline phases of hydrated cement or is absorbed into the C-S-H gel or crystals [26]. In contrast, when increasing the PRB at weight percentage of 15.0% level of mix, a fluctuation in Pb leachability was detected probably due to carbonation by atmospheric CO₂ which lowered pH of S/S system leachant [28]. The addition of 15% PRB has caused unexpectedly a pH to be close to 8.0 after 24 hours, and

hence, the Pb leachability has been increased and this can be attributed to the presence of CaSO_4 (15.32%) in the PRB structure.

At neutral and alkaline pH are for sufficient carbonate present environment, the species accounted for a significant fraction of soluble inorganic Cu in solution is the soluble CuCO_3 formed due to carbonation process and it was not pH dependent [29]. All the mixes reduce Zn leachability less than the proposed criteria of 5 mg/L approved by US EPA for TCLP standards (table 7). This indicates that s/s technology suitable for HMs treatment and the PRB is good as second binder. TCLP test, the 15% level of mix had the lowest leachate Zn concentration < 0.003 ppm (TCLP test). The leachate Ni concentration attained at less than the proposed criteria of 7.0 mg/L approved by US EPA for TCLP standards (Table 7). They are accounting for 95.7%, 97.4% and 97.0% of (Ni) leachate concentration respectively. However, 5% Cr concentration is acceptable for TCLP standard (Table 7). Chromium (VI) (chromates) must be reduced to chromium (III) for stabilization within the S/S system [30].

4-CONCLUSION

Based on the above studying carried out on the heavy metals that compound the drilling mud results showed that:

Leaching of metals from the s/s products in a distilled water batch extraction was affected by Porotherm red bricks addition.

Unconfined compressive strength was increased by Porotherm red bricks increments.

The grinding of the monolith decreases the pH of the leachate and not affected by time.

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