Studies on factors affecting unconfined compressive strength of industrial rubber sludge containing heavy metals treated using ordinary Portland cement via stabilization/solidification technique

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Abstract. High concentration of selected heavy metals within industrial rubber sludge collected from rubber industry wastewater treatment plant has classified the waste as scheduled waste. Special treatment to the waste by using ordinary Portland cement via solidification/stabilization (S/S) technique has been performed in laboratory scale. The objective of this research is to determine related factors that affect unconfined compressive strength (UCS) performance of stabilised/solidified (s/s) cube specimens which contains industrial rubber sludge waste. Other parameters observed include the curing condition (i.e. air and water immersion curing method), waste composition, specimen age and density. The prepared fresh mix were cast in plastic moulds in order to produce 50 mm³ cubical shape specimens and leaved to set approximately 24 to 48 hours. The prepared specimen batches are S1 (90% OPC + 10% waste), S2 (70% OPC + 30% waste), S3 (50% OPC + 50% waste). UCS was performed on respective specimen age of 7 and 28 days. Positive results were obtained as relatively the average compressive strength of 7 day air cured specimens reach 5.25 MPa, 5.28 MPa, and 2.16 MPa for S1, S2 and S3. While, 28 days air cured specimens results are 9.59 MPa, 8.01 MPa, and 1.46 MPa for S1, S2, and S3 respectively. As for water immersion, the compressive strengths are 8.19 MPa, 4.93 MPa, and 1.90 MPa for 7 days, and 7.75 MPa, 10.10 MPa, and 2.11 MPa for 28 days at respective S1, S2 and S3 sequence. As conclusion, the specimens prepared passed the minimum requirement for secured landfill disposal which is at 1 MPa.

1. Introduction

Industrial rubber sludge waste has taken an attention as the increased in rubber manufacturing production also generates closed to 240 tonnes of scheduled waste each year [1-2]. In general, the increased volume of waste generated also created other problems associated with handling and storage of the waste prior to disposal [3].

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Serious toxicity problem especially to human and other living creature (i.e. animals and plants) could possible occur as if the waste were exposed to rain or water runoff which act as transport media of contaminants as the migration of contaminants can starts from drainage system and into the water bodies (i.e. river and/or groundwater) [4]. As waste hazard potential is acknowledged due to heavy metals toxicity, an approach to treat the waste via stabilisation and solidification (S/S) technique in laboratory scale was conducted.

Fundamentally, stabilisation is a technique which chemically reduces the waste hazard potential by altering the properties of the contaminants into less soluble, mobile and toxic [5]. Solidification is a technique which involves physical process that encapsulates the waste into solidified form or material. The difference as compared to stabilisation technique is that solidification process does not necessarily involve a chemical interaction between the contaminants and solidifying additives [6]. The concern particularly focused on the effectiveness this technique (i.e. stabilisation and solidification) to immobilise the waste contaminants in an inert form using Ordinary Portland cement as primary binder. As curing condition plays a significant role in S/S, the objective of this work is to determine the unconfined compressive strength (UCS) performance of stabilised/solidified which contains industrial rubber sludge waste. The initial and final strength basically corresponds to the effectiveness of the S/S treatment whereby a minimum strength of 1 MPa should be achieved in order for the stabilised/solidified specimens are to be disposed in a sanitary or secured landfill [6].

2. Materials and methods

2.1 Preparation of fresh mix

The fresh mixes were prepared according to factorial design method whereby each ingredient (i.e. unit of kilograms) was calculated in order to achieve desired ratio of ordinary Portland cement (OPC) and waste in each specimen batch (refer to Table 2). Table 1 shows the factorial design for control batches (i.e. OPC and water only) at different water to cement (w/c) ratio of 0.33 and 0.50.

| Batch ID | Composition | OPC (kg) | Waste (kg) | Water (kg) | Water-cement (w/c) ratio |
|----------|-------------|----------|------------|------------|-------------------------|
| CB1      | OPC + water | 4.545    | 0.0        | 1.500      | 0.33                    |
| CB2      | OPC + water | 4.545    | 0.0        | 2.273      | 0.50                    |

| Batch ID | Composition | OPC (kg) | Waste (kg) | Water (kg) | Water-cement (w/c) ratio |
|----------|-------------|----------|------------|------------|-------------------------|
| S1       | 90% OPC + 10% waste | 4.770    | 0.530      | 1.860      | 0.4                     |
| S2       | 70% OPC + 30% waste | 3.710    | 1.590      | 1.692      | 0.5                     |
| S3       | 50% OPC + 50% waste | 2.650    | 2.650      | 1.782      | 0.6                     |
As the ingredients were carefully weighted and transferred into the mixer, the dry mixture of OPC and waste were mixed well prior to addition of water. Specific amount of water added into the mixture in order to achieve desired water to cement (w/c) ratio. This is because too much water will affect the compressive strength of the stabilised/solidified specimen where w/c ratio should be around 0.3 to 0.6 [7]. Fundamentally, water is added until certain consistency was achieved, whereby an excessive of water generally will produced more porous specimen as the setting or hardening completed [6].

The fresh mix was transferred into mould in order to cast and produce 50 mm$^3$ cube stabilised/solidified specimen. The size of 50 mm$^3$ was chosen due to minimise the waste generation in testing [6] whereby these specimens were classified as treated hazardous waste. The moulds were vibrated at approximately 15 s in order to compact and remove any remaining air bubbles trapped in the fresh mix. In each batch, approximately 20-25 cube specimens can be produced. The moulds were placed at dry and dark room to allow hardening process of the fresh mix. The hardening process took approximately 24 hours to completely hardened prior to demould process (i.e. removing the specimens from the moulds) can be performed [7].

As the demould process performed, the specimens were stored in a closed large plastic bag equipped with some wet tissue to preserve high humidity in order to continue the hydration reaction [6].

2.2 Unconfined compressive strength test

UCS test were conducted at specific specimen age of 7 days for determining the initial strength and 28 days for final strength according to BS EN 196-1:2005 [6]. Prior to testing at specific specimen age (i.e. 7 and 28 days) triplicate of cube specimen were immersed in water for approximately 24 hours before the testing day as these cube specimens will represent the immersed cube specimen. While the dry or air cured specimens were taken directly from the storage bag. By means at every UCS testing, there are triplicate of dry and also immersed cube specimens (i.e. total of six (6) cubes produced: three dry and three immersed cubes). The concrete compression machine was calibrated according to same standard (i.e. BS EN 196-1:2005) used to perform the UCS test [6]. An average reading from triplicates specimen cube compressions was taken as the final result for each batch.

3. Results and discussion

The initial and final strength of stabilised/solidified (s/s) specimens depends on a few factors (i.e. specimen age, waste composition, density and also curing methods) [6]. In order to evaluate the specimen batches prepared, control batches which are CB1 (w/c ratio: 0.33) and CB2 (w/c ratio: 0.5) were prepared and tested. Control batch represent the baseline reference for comparison purposes in order to identify the effect of organic waste composition with respect to strength, density, water-to-cement (w/c) ratio of the specimens.
3.1 Effects of air curing condition on initial and final strength

Air curing is one of the essential methods in hardening process of the stabilised/solidified (s/s) specimen due to prevent contaminants from leaching out from the matrix [6] whereby these specimens were sealed in large plastic bag together with some wet tissue to keep the moist environment and also to exclude carbon dioxide which could affect the alkalinity of the s/s specimens [6]. The aim is to compare the performance of UCS in the presence of industrial rubber sludge at three different compositions (i.e. 10% waste in S1, 30% waste in S2 and 50% waste in S3). The UCS 7 day analyses of air curing specimens showed in Figure 1 illustrates almost linear decreasing of the initial strength from S1 to S3. The collapse trend of initial strength showed is clearly due to the increased of organic waste composition (i.e. industrial rubber sludge) added into OPC. Combination of OPC as an inorganic material with organic waste has a certain percentage limitation up to not more than 30% [6].

The UCS test performed at 28 days of air cured specimens is illustrated in Figure 2, which represent as the final strength of control and sample batch specimens. The analyses in Figure 2 indicate that there are strength increment occurred especially on the both control batches and also in S2 sample batch. This occurrence is normal towards the control batches because it do not contains any waste or impurities which can affect the hydration process [6], but an increase in S2 sample batch revealed the acceptable and also compatibility of the OPC matrix (as an inorganic component) to accept the organic waste loading within the cement structure. The continual of strength development progress of S2 batch as can be seen from 7 days UCS in Figure 1 to 28 days UCS in Figure 2 shows the optimum amount of organic content which can be treated with OPC via stabilisation/solidification technique.

However, specimen batch S1 and S3 in Figure 2 showed decreasing in strength as compared to its initial strength (i.e. Figure 1). The composition of waste in S1 was at minimum (i.e. 10% waste) and does not affect much on the strength development. Clearly the strength of the specimen was truly affected in S3 sample batch which contains approximately 50% of waste. Too much of the organic waste does affect the rigidity and also compactness of the specimens because both materials (i.e. inorganic and organic component) do not mixed well due to the formation of heterogeneous specimen in S3 batch.

By comparing the water-to-cement (w/c) ratio between the control batches and specimen batches in Figure 1 indicates higher water content contributes to lower strength development at initial strength. The highest w/c ratio is S3 batch (w/c ratio: 0.6), followed by S2 (w/c ratio: 0.5) and S1 with lowest w/c ratio of 0.4. The findings also demonstrates that the presence of organic waste (i.e. rubber sludge) have eventually increased the w/c ratio and leads to low strength of the specimens produced. The absence of waste such as in both control batches generally does not affecting the growth of strength in initial and final strength as shown in both Figure 1 and 2.
3.2 Effects of water immersion curing condition on initial and final strength

Water immersion curing method has also been applied for 7 and 28 days UCS which can be clearly observed in respective Figure 3 and 4. In this water immersion curing method, triplicate of hardened specimens were immersed in water at 24 hour before the UCS test. The purpose of the water immersion was to prove that water does enhance the hydration process which in return aiding the strength development of the specimens and not just normal drying [8]. Water is an essential component in cement hydration reaction whereby it assist the growth of calcium-silicate-hydrate (C-S-H) at initial strength [9].

The formation of C-S-H usually persistent as the specimen age increased as this will contribute to specimen strength development. This can be observed in Figure 4 as the final strength of all specimen batches at 28 days increased as compared to its initial strength at 7 days in Figure 3. S2 specimens show quite remarkable average final strength as compared to S1 and S3 batch specimens at 28 days. The findings (i.e. Figure 4) show the compatibility between the organic waste and inorganic material of OPC with the ability to produce higher strength at 28 days.

However in S/S treatment, water immersion can lead to leaching of contaminants within the specimen [6]. This is because water is able to penetrate into the specimen through the voids of the micro-capillaries present in the specimen which was produced during hydration process. Due to that only, air curing method was applied in this S/S treatment. Comparison between air curing method (i.e. Figure 1) and water immersion (i.e. Figure 3) of 7 days sample specimens (i.e. S1, S2, and S3 specimens) indicates that initial strength was found to be lower in water immersion. The reduction of initial strength was caused by water intrusion into the micro pores within the specimens leading to excess water in the specimens. At the initial strength stage, hydration reaction still occur but in the presence of excess water, more micro-capillaries formation were produced during the process leading to the development of more porous specimen. As the specimen’s porosity increased, the strength of the specimen was inversely decreased [10-12].

Water immersion curing method also been applied to evaluate the stability of specimens under water immersion [8]. The stability of specimens under water immersion also examines matrix dissolution or detrimental swelling which can disrupt the physical of the specimen [6]. Observations of the immersed specimens’ condition were performed prior to UCS test. Based on the findings appeared on Figure 3 and 4, both revealed that all specimens are stable in water immersion and able to show significant compressive strength via UCS test throughout respective 7 and 28 days.
3.3 Correlation between waste compositions, unconfined compressive strength and density

The correlation of initial strength and density of air cured specimens are demonstrated in Figure 5. Density of the specimen also correlated with the water-to-cement (w/c) ratio and its strength. Analyses on Figure 5 revealed that the density does slightly reduced when comparing both control batches, as the highest initial strength was obtained at the lowest w/c ratio batch (i.e. CB1). As w/c ratio increases, the strength and density decreases [10-11]. The water demand occurred especially in the presence of other components such as organic waste (i.e. rubber sludge). Observation on all specimen batches (i.e. S1, S2 and S3 batches) clearly demonstrated that there was an increased of w/c ratio as the amount of waste was increased. The increment of w/c ratio was directly related to the extra water needed to provide sufficient consistency to the fresh mix preparation. As the organic waste compositions were increased, the strength and density also relatively decreased.

The amazing thing of this rubber sludge waste was that, even at 50% waste was added into the OPC, the initial strength of S3 batch specimens (Figure 5) were still capable to surpass the minimum disposal limit required at 1 MPa, whereby an average strength of triplicate testing produced 2.16 MPa was obtained.

Observation on specimen S2 batch on both 7 days (Figure 5) and 28 days (Figure 6) revealed that there was an increased in strength (i.e. initial to final strength). The rubber sludge at 30% in S2 reacts positively with OPC and was able to produce sufficient strength at 28 days. One of the characteristics of rubber sludge was its ability to absorb and retain water in its matrix, whereby these water were then consumed by OPC to enhance hydration reaction which resulting an increment in specimen strength. Contrary, this occurrence was not appeared either in S1 or S3 batches in Figure 6. Justification was that S1 contains quite low amount of rubber sludge (i.e. 10%) and the overall strength performance was governed by OPC as the majority component in the S1 specimen. Whereas, S3 specimens consist approximately 50% of rubber sludge and this was too much for the OPC matrix to sustain strength whereby good compatibility as the organic component should not equivalent to the OPC composition. This has been supported by previous studies which highlight the limitation of organic waste that can be added into OPC in Stabilisation/Solidification (S/S) treatment technique [6].

Analyses on Figure 6 revealed that both control batches (i.e. CB1 and CB2) shows a positive increment in final strength as compared to its initial strength in Figure 5. However, the final strength of specimen batches which contains rubber sludge as in S1, S2 and S3 have revealed to be affected. The changes in the final strength somehow did some small changes in the density of specimen batches which can be clearly seen in Figure 6. However, overall final strength performances of all specimen batches were found to be sufficiently above the minimum disposal limit at 1 MPa.

![Figure 5: Initial strength and density correlation of air curing specimens](image1)

![Figure 6: Final strength and density correlation of air curing specimens](image2)
4. Conclusion

Based on this study, it can be concluded that unconfined compressive strength (i.e. initial and final strength) and density of specimen batches can be affected by several factors such as organic waste composition, specimen age and curing conditions (i.e. air and water immersion curing method). The optimum condition revealed that OPC was able to react positively up to 30% of rubber sludge composition.

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