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EXPLORATORY STUDY ON THE MECHANICAL AND PHYSICAL PROPERTIES OF CONCRETE CONTAINING SULFUR

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Graphical abstract

Abstract

This research is an exploratory experiment into sulfur concrete used not as a complete replacement of cement but as an additional material in percentage of the cement content. The aim of this research was to explore the possible appreciation of mechanical and physical properties of concrete containing sulfur with percentages of 1%, 5% and 10% of the cement content. The sulfur used here was not heat-activated, hence the binding effect in sulfur was absent. The experimental results revealed that concrete containing sulfur did not perform better in their strength properties, both compressive strength and flexural strength. The physical properties such as water penetration and water absorption for concrete containing sulfur also showed poor performance in comparison to ordinary Portland cement concrete. Such phenomena are very likely due to the sulfur not being activated by heat. Carbonation test did not show good results as a longer term of testing is required. Drying shrinkage property was found to be encouraging in that concrete containing 10% sulfur had quite significant reduction in drying shrinkage as opposed to ordinary Portland cement concrete.

Keywords: Sulfur concrete; mechanical and physical properties.

Abstrak

Kajian ini merupakan kajian awal ke atas konkrit sulfur yang mana sulfur digunakan sebagai bahan tambah berdasarkan peratusan kepada kandungan simen. Kajian ini berbentuk eksplorasi untuk mengkaji samada terdapat peningkatan dalam sifat mekanikal dan fizikal bagi konkrit yang mempunyai kandungan sulfur 1%, 5% dan 10% kepada kandungan simen. Sulfur yang digunakan dalam kajian ini tidak dipanaskan, maka sifat lekatan sulfur itu tidak diwujudkan. Hasil kajian ini mendapati tidak ada peningkatan sifat mekanikal dan fizikal dalam konkrit sulfur. Pendapatan ini mungkin disebabkan oleh reaksi sulfur yang tidak diaktifkan. Ujian pengkarbonatan tidak menunjukkan hasil yang memuaskan kerana ianya memerlukan tempoh ujian yang lebih panjang. Ujian pengecutan disebaliknya menunjukkan konkrit yang mengandungi 10% sulfur menghasilkan pengecutan yang menurun dibandingkan dengan konkrit simen Portland biasa.

Kata kunci: Konkrit sulfur; sifat mekanikal dan fizikal

1.0 INTRODUCTION

Sulfur is a byproduct of petro-chemical refinery and liquefied natural gas production processes [1] also known as involuntary sulfur. It has been reported that a gas resource in Qatar produces an average of 12,000 tons of sulfur per day [2]. Sulfur is a non-metallic chemical element denoted as S with bright yellow crystalline solid appearance when at room temperature. It is the thirteenth most abundant element in the earth crust [3]. The application of sulfur includes agricultural industry to manufacture phosphates, nitrogen and sulfate fertilizers. Other sulfur related industries are synthetic rubber vulcanization, water treatment, pesticides, personal care products and cosmetics [4].

In an attempt to re-use this sulfur waste for construction, history revealed that sulfur has been utilized in concrete mixing as early as 1920s. Some issues found in this early use were durability problems when subjected to repeated freezing and thawing cycles, water immersion and humid conditions [5]. Such circumstances are potentially caused by internal stresses within the crystalline structure of the concrete [6]. More research in the 1970s has led to the use of modified sulfur in concrete that could eliminate the aforementioned problems. This has further developed into the use of sulfur concrete on a commercialized scale [1]. Nevertheless, because such application depends very much on technological expertise and strong economical reasons, sulfur concrete has found its place only in niche applications, and thus very limited.

This application of sulfur in concrete refers to a complete replacement of cement and water. In conventional concrete, cement and water are used to produce the binding effect in the concrete mix. In sulfur concrete, cement and water are not required. The sulfur is heated up to 150°C to activate the binding effect in the mix and is prepared in four stages [7]. Sulfur acts as a thermoplastic material that binds the non-reactive aggregates together. The aggregate used is generally coarse aggregate made of gravel or crushed rock and fine aggregate such as sand. The composition generally consists of 78 to 88 weight percentage of aggregate and 12 to 22 weight percentage of sulfur. The sulfur might contain 5% of plasticizers to increase to workability [8].

The special advantage of sulfur concrete lies in the following exhibited characteristics: high corrosion resistance for both biological and chemical corrosion (ie. protection against acid and salt attack), fast hardening (ie. very early strength development without needing prolonged curing as in normal concrete as shown in Figure 1), greater strength properties, and low permeability [9] [10]. A general comparison of various properties between sulfur concrete and ordinary Portland cement concrete are presented in Table 1, a study conducted by different laboratories and reported by [11]. As such, sulfur concrete is advantageous for applications in corrosive environments such as for structures at the seaside and for repair applications because of fast hardening behavior [7] [12].

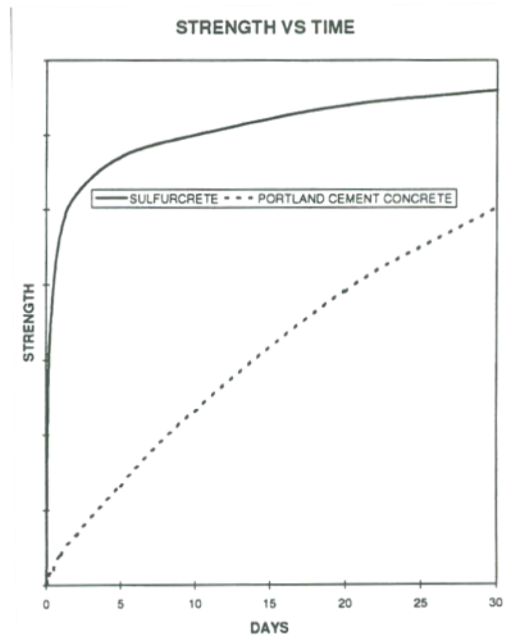

Figure 1 Strength development between sulfur concrete and ordinary Portland cement concrete [4]

Table 1 Properties of sulfur concrete compared to ordinary Portland cement concrete [11]

Property	Compared with 34.5 MPa Portland cement concrete
Abrasion resistance	Much greater
Bond strength to concrete	Much greater
Bond strength to reinforcing steel	Greater
Coefficient of linear expansion	Equivalent
Compressive creep	Less
Compressive strength	Greater
Corrosion resistance	Much greater
Durability under thermal cycling	Equivalent of higher
Fatigue resistance	Much greater
Fire resistance	Slightly less
Flexural strength	Greater
Modulus of elasticity	Greater
Splitting tensile strength	Greater
Thermal conductivity	Less
Water permeability	Much less

A patented and already commercialized sulfur concrete claimed a compressive strength ranging between 40 to 65 MPa and 8.4 to 11.2 MPa for flexural strength depending on the mix design [4]. Works by [12] on sulfur mortar containing 30% sulfur binder produced compressive strength and flexural strength of over 70 MPa and over 12 MPa, respectively. For sulfur concrete with 15% sulfur binder, a compressive strength ranging 50 to 60 MPa was reported while the flexural strength ranges between 8 to 10 MPa. Other properties found by [12] are presented in Table 2.

Table 2 Properties of sulfur mortar and sulfur concrete by [12]

Properties	Sulfur mortar	Sulfur concrete
Compressive strength (MPa)	70-75	50-60
Flexural strength (MPa)	12-13	8-10
Indirect tensile strength (MPa)	5-6	5
Shrinkage (mm/m)	0.6-0.7	1.4

Figure 2 Specimens in mould after casting

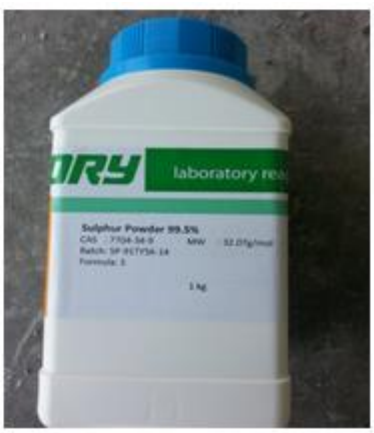

Figure 3 Sulfur powder in packaging of 1 kg bottle

To date, no publications or commercial articles have been found mentioning the use of sulfur concrete in Malaysia, both in the research arena or in the construction industry. The main objective of this paper is to present findings from an exploratory research conducted on sulfur concrete. The aims of this exploration are to determine any form and pattern of appreciation with regards to the mechanical and physical properties of sulfur concrete as opposed to conventional concrete. The mechanical properties that have been investigated are compressive strength and flexural strength, while the physical properties include concrete carbonation, drying shrinkage, water penetration and water absorption.

In the context of this research, the sulfur used to produce sulfur concrete is not a total replacement of cement and water. This approach requires expert technology and stringent control in heating up the sulfur to a designated temperature such that it behaves as a binder. For this reason, this approach is not yet exploited here. In this research, the sulfur used is referred to as an additional material, an admixture in terms of percentage of the cement content in the concrete mix. The selected percentages of sulfur are 1%, 5% and 10% of the cement content. More description on the mix design is given in the experimental chapter of this paper. The conventional concrete used refers to normal weight concrete using ordinary Portland cement with 2400 kg/m^3 density and 0% sulfur acting as the control specimen.

2.0 EXPERIMENTAL

The experimental setup was divided into two categories. The first category was to determine the mechanical properties namely compressive strength and flexural strength. The second category was to investigate the physical properties such as carbonation, drying shrinkage, water penetration and water absorption.

In the first category, for compressive strength test, cube size 100 × 100 × 100 mm was used and rectangular prism size 100 × 100 × 600 mm for flexural strength test. A total of four variations of concrete mix, ie. 0%, 1%, 5% and 10% sulfur as explained in the preceeding section, with 2 test specimens for each percentage is illustrated in Table 3. In the second category, for water penetration and water absorption test, a 150 × 150 × 150 mm specimen size was used. For drying shrinkage test, a 110 mm diameter × 300 mm high cylinder was used. For carbonation test, cube size of 100 × 100 × 100 mm was used. The distribution of specimen numbers and concrete mix are similar to that of in the first category, ie. 0%, 1%, 5% and 10% sulfur and 2 number of specimens for each variation and type of test, except for drying shrinkage test which has 3 number of specimens. This distribution is given in Table 4. All the specimens are shown in Figure 2. The sulfur used comes in 1 kg bottle and was purchased from a local chemical supplier as shown in Figure 3.

Table 3 Number of specimens for test on mechanical properties

Concrete mix with percentage of sulfur	Compressive strength test		Flexural strength test
	At 28 day	At 56 day	At 28 day
0%	2	2	2
1%	2	2	2
5%	2	2	2
10%	2	2	2

Note: A total of 24 specimens.

The test methods for compressive strength and flexural strength are based on BS EN 12390-3: 2009 [13], and BS EN 12390-5: 2009 [14], respectively. The test procedures for water penetration, water absorption and drying shrinkage are referenced to BS 12390-8: 2009 [15], BS 1881-122: 2011 [16] and BS ISO 1920-8: 2009 [17], respectively. For drying shrinkage test, some modifications of specimen size and testing frame were made to accommodate with the available materials. A cylinder type specimen was used instead of a prism and a vertical standing test frame applied instead of a horizontal comparator as proposed in BS ISO 1920-8: 2009 [17]. The drying shrinkage test setup using a vertical frame with the cylindrical specimen placed permanently immediately after demoulding and a dial gauge mounted on the top is illustrated in

Figure **4**.

The targeted strength for the concrete mix is between 25 to 30 MPa and the density is 2400 kg/m^3. The mix proportion for 1 m^3 of concrete used in this study is presented in Table 5. The water-cement ratio applied was 0.57.

Figure 4 Drying shrinkage test setup using vertical frame with permanent dial gauge

Figure 5 Adding sulfur powder into concrete mix

Table 4 Number of specimens for test on physical properties

Concrete mix with percentage of sulfur	Water absorption and water penetration test		Carbonation test				Drying shrinkage
			Air-con room env.		Ambient room env.		
	28 d	56 d	28 d	56 d	28 d	56 d	
0%	2	2	2	2	2	2	3
1%	2	2	2	2	2	2	3
5%	2	2	2	2	2	2	3
10%	2	2	2	2	2	2	3

Note: Total number of specimens is 60.

Table 5 Mix proportion for 1 m^3 of concrete with 0.57 w/c ratio given in mass of material (kg)

Concrete mix with percentage of sulfur	Cement	Fine agg.	Coarse agg.	Sulfur
0%	300	868	1061	0
1%	300	868	1061	3
5%	300	868	1061	15
10%	300	868	1061	30

In order to obtain genuine result and to minimize possible variations in the result analysis, no other additional admixtures such as water reducer or superplasticizer are used. Figure 5 shows addition of sulfur powder into the mix. All mixes are mixed manually with hand for better control as presented in Figure 6. Both fine and coarse aggregates are sieved to obtain the required size and after air dried prior to mixing. The fine aggregate used were no greater than 5 mm and coarse aggregate between 5 to 20 mm in size. Figure 7 shows the preparation of the raw materials

Figure 6 Manual mixing of concrete with sulfur content

Figure 7 Preparation of raw materials such as coarse aggregate

3.0 RESULTS AND DISCUSSION

3.1 Compressive Strength And Flexural Strength

The average density of the specimens is in the range of 2310 to 2357 kg/m^3. Figure 8 and Figure 9 shows the compressive strength test and flexural strength test performed in the laboratory, respectively. The result of compressive strength at 28 day and 56 day is presented in Table 6 and Figure 10. On the contrary to the findings of [1], [4], [7] and [11] for both modified and unmodified sulfur concrete which claimed that the strength of sulphur concrete is greater than that of ordinary Portland cement concrete, this research found that the compressive strength of concrete containing sulfur is lower than that of ordinary Portland cement concrete. At 28 days, ordinary Portland cement concrete achieved a compressive strength of 42.4 MPa and concrete with 10% sulfur content appeared to have compressive strength of 21.5 MPa, which is half of the ordinary Portland cement concrete. The strength increment pattern from 28 day to 56 day for concrete containing sulfur does not differ from that of the ordinary Portland cement concrete. It is also observed that the more sulfur added into the concrete mix, the lesser the compressive strength.

For flexural strength, similar reduction trend is not observed apart from concrete with 10% sulfur which exhibited 3.9 MPa as opposed to an average of 5 MPa in the other specimens including the ordinary Portland cement concrete. The results are tabulated in Table 6 and illustrated in Figure 11.

The sulfur concrete in this experiment failed to show positive strength performance. Instead, there was negative strength behaviour observed. This is likely due to the fact that the sulfur in this laboratory testing was added into the concrete mix without being heated or boiled as practiced by other researchers of sulfur concrete [7]. It is believed that the absence of heating did not activate the binder effect in sulfur. As such, the hardening of concrete and hydration process only depended on the cement component. It is also possible that the presence of sulfur together with cement produces a negative chemical reaction which could possibly affect the cement hydration causing a decrease in strength development in concrete containing sulfur. In order to confirm such hypotheses, further research in the chemical component and microstructure of cement reacting with sulfur may help. Further research on adding boiled sulfur into the concrete mix as compared to adding sulfur powder may also be useful to learn whether heating up of sulfur does activate the bonding reaction

Figure 8 Compressive strength test

Figure 9 Flexural strength test

Table 6 Tabulated result of mechanical and physical propertie
s

Concrete mix with percentage of sulfur	Compressive strength (MPa)		Flexural strength (MPa)	Water penetration (mm)		Water absorption (%)		Drying shrinkage (microstrain)				Carbonation depth (mm)			
												Aircon env.		Ambient env.	
	28d	56d	28d	28d	56d	28d	56d	1d	7d	28d	56d	28d	56d	28d	56d
0%	42.4	47.3	5.2	88	90	5.7	5.9	27.8	126.7	248.9	293.3	0	0.5	0.5	1.5
1%	39.3	41.8	5.5	139	122	6.6	6.3	26.7	93.3	198.9	232.2	0	0	1	1.5
5%	25.8	31.6	5.3	150	132	7.1	7.3	20.0	100.0	197.8	227.8	0	0	1.5	2
10%	21.5	24.6	3.9	150	150	6.3	6.4	21.1	78.9	168.9	190.0	0	0	0.5	1

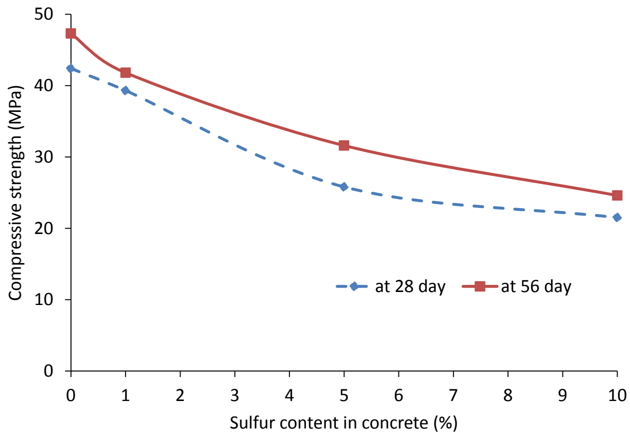

Figure 10 Compressive strength of concrete containing sulfur at 28 day and 56 day

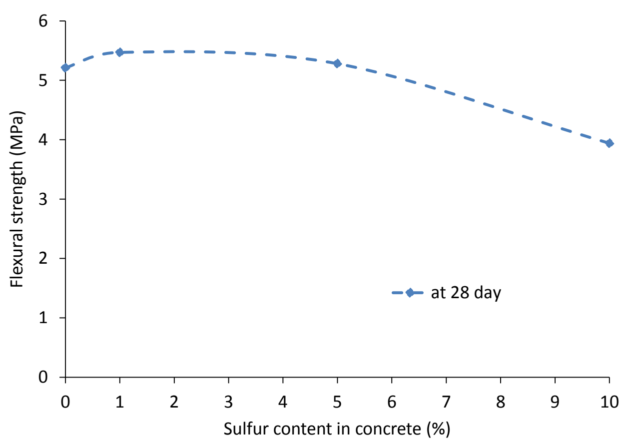

Figure 11 Flexural strength of concrete containing sulfur at 28 day

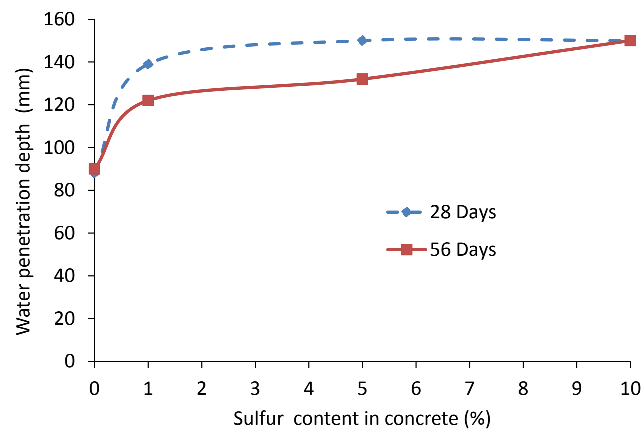

Figure 12 Water penetration depth of concrete containing sulfur at 28 day and 56 day

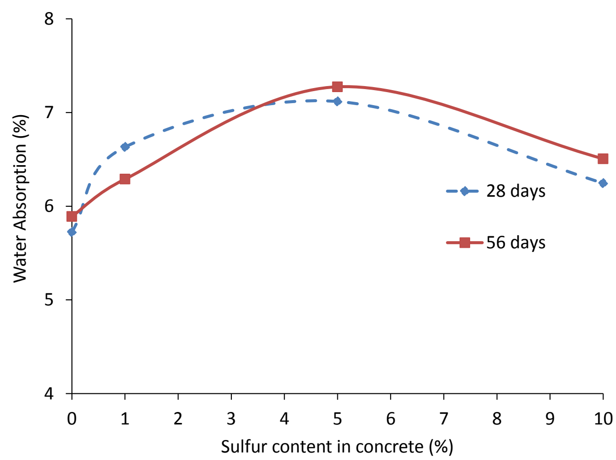

Figure 13 Water absorption of concrete containing sulfur at 28 day and 56 day

3.2 Water Penetration And Water Absorption

The results of water penetration and water absorption are presented in **Table 6**. The graphical presentation of the results is given in **Figure 12** and **Figure 13** at 28 day and 56 day for water penetration and water absorption respectively. **Figure 14** shows the apparatus set up with 3 cube specimens applied to a water pressure of 5 bars or 500 kPa and **Figure 15** shows a halved cube for the measurement of water penetration depth.

Results showed that depth of water penetration for concrete containing sulfur did not improve, instead worsen with 150 mm , ie. full depth, for 10% sulfur concrete compared to approximately 90 mm depth of the control specimen, ie. 0% sulfur concrete. For water absorption, inconsistent absorption percentages were observed for sulfur concrete, ie. more than 6%. Nevertheless, they were all greater than the control specimen, ie. less than 6%. Again, such negative performance could likely be due to the non-activated sulfur binding effect caused by the absence of sulfur heating.

Figure 14 Water penetration test setup up to water pressure of 5 bars or 500 kPa

Figure 15 Control cube specimen halved to measure water penetration depth

3.3 Drying Shrinkage

Figure 16 shows the drying shrinkage results measured from 0 day up to 56 days for concrete containing sulfur compared to the control specimen with 0% sulfur. These results are also tabulated in Table 6. It is observed that concrete containing sulfur does shrink less compared to the ones without sulfur content. This provided a positive performance. A reducing pattern of shrinkage is seen when higher sulfur content is applied.

At 28 day, concrete with only ordinary Portland cement resulted in a drying shrinkage of 248.9 microstrain, while concrete containing 10% sulfur measured a drying shrinkage of 168.9 microstrain,which is approximately 30% lesser. At 56 day, the reduction trend increased showing an approximately 40% reduction in drying shrinkage when compare ordinary Portland cement concrete (293.3 microstrain) to concrete with 10% sulfur content (190 microstrain

When compared to other physical properties such as water penetration and water absorption which produced a down-trend performance, concrete containing sulfur exhibited an encouraging behavior for drying shrinkage property. This is quite likely because the reaction between sulfur and cement causes less water being used up in the hydration process, hence, lowering the drying shrinkage.

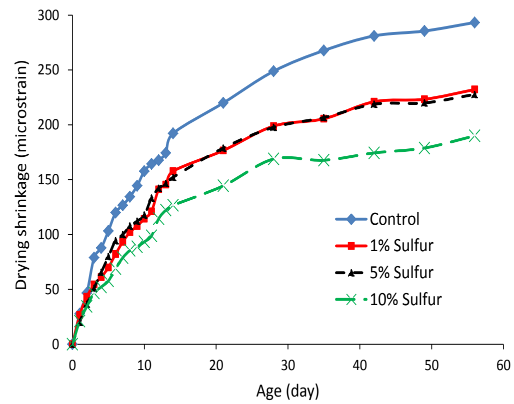

Figure 16 Drying shrinkage of specimens from 0 day to 56 day

Figure 17 Relative humidity and temperature hand-held tool kit

Figure 18 Relative humidity and temperature hand-held tool kit

Figure 19 Spray of phenolphthalein solution onto a halved cube

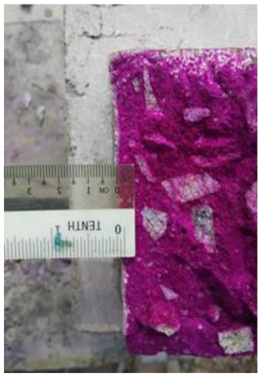

Figure 19 Measurement of carbonated depth, ie. non purple

3.4 Carbonation

The study of carbonation property involved monitoring of the temperature and relative humidity for the two different environments as highlighted in Table 4, ie. indoor air conditioned environment and sheltered ambient environment. A relative humidity and temperature hand-held tool kit as shown in Figure 17 was used for the purpose of monitoring. Table 7 and Table 8 present the relative humidity and temperature of the two environments, sheltered ambient environment and indoor air conditioned environment, respectively, used to store specimens for carbonation study purposes.

In order to check the carbonation depth of concrete after being conditioned under both air conditioned and sheltered ambient environment, both at 28 day and 56 day, Phenolphthalein solution was used (**Error! Reference source not found.**). Phenolphthalein solution is sprayed onto the halved surface of the cube as illustrated in Figure 19. Surface that is not carbonated result in purple colour while carbonated surface remained colourless, being the surface that is measured (**Error! Reference source not found.**). This method depends solely on pH change and therefore the influence may not be clearly identified if carbonation only occurs partially, or if carbonation occurs but with pH beyond the scope changeable by the indicator. Nevertheless, such simplest and easiest method in a visual aspect for testing carbonation is quite widely used at construction sites, and its use may not be restricted in reality.

Carbonation is the formation of calcium carbonate ($CaCO_3$) by a chemical reaction in the concrete. The chemical reaction needs 3 important materials which are carbon dioxide, water and cement hydration products. Carbon dioxide in the air penetrates into non-saturated pores of concrete to form calcium carbonate and water.

Table 7 Average relative humidity and temperature for sheltered ambient environment

Time	Relative humidity (%)	Temperature (°C)
Morning	73.2	31.5
Afternoon	77.7	29.8
Night	84.2	27.5

Table 8 Average relative humidity and temperature for indoor air conditioned environment

Time	Relative humidity (%)	Temperature (°C)
All time	64.9	24.5

Concrete has high alkalinity of about pH 12 to pH13 when mixed, due to a large formation of calcium hydroxide, $Ca(OH)_2$. Calcium hydroxide does not contribute to strength development but it acts as a protective layer for steel reinforcement. This protective layer will be destroyed when the alkalinity reduces to pH 9 as a result of contact with carbon dioxide [18].

The pH effect can be pictured as 3 different categories which are (1) no corrosion, passivation from pH 14 to pH 9.5, (2) passivation decreases, corrosion starts from pH 9.5 to pH 5.5 and (3) the rapid and heavy corrosion from pH 5.5 to pH 1. Carbonation will affect the cement hydroxide product (calcium hydroxide → calcium carbonate) causing a decrease of alkalinity from pH 12.6 to pH 8 where the reinforcement will start to experience corrosion.

The carbonation process requires the presence of water to let carbon dioxide to dissolve forming H_2CO_3. For a low relative humidity of less than 50%, the diffusion of carbon dioxide into concrete is high but there is not enough water in the pores to generate carbonation. On the other hand, for a high relative humidity the diffusion of carbon dioxide is very low also, reducing the carbonation rate. Therefore, the optimum relative humidity value between 40% and 70% is use in majority of the research on concrete carbonation [19].

There are two parameters to alter the speed of carbonation, namely porosity of the concrete and the moisture content or relative humidity of the concrete. From the described mechanism, carbon dioxide in the air is needed to sustain the carbonation process. Pores in concrete allow carbon dioxide to penetrate into the concrete and cause carbonation. Therefore, increase in porosity will lead to increase in permeability and increase the rate of carbonation. But if porosity decreases, permeability and rate of carbonation will also decrease.

The result of carbonation study in Table 6 shows an inconclusive measurement, with carbonation depth of 0 mm at 28 day for all specimens, and 0.5 mm to 2 mm range for both control specimen and specimen containing sulfur in both air conditioned and sheltered ambient environment. Because of the inconsistency, no conclusion can be made with regards to carbonation. Nonetheless, it is expected that sulfur concrete has the ability to reduce carbonation because of low permeability and low porosity when the sulfur is heated and activated. For the purpose of carbonation study, it is recommended that a longer term be applied in order to yield reliable result.

4.0 CONCLUSION

Sulfur concrete has been used as early as 1920s and commercially available in the 1970s. The use of sulfur concrete aimed at re-using sulfur waste, a byproduct of petro-chemical and liquefied natural gas refinery process. There are many advantages in the use of sulfur concrete. One main advantage is that sulfur concrete creates a high corrosion resistance apart from providing greater mechanical properties when compared to ordinary Portland cement concrete. The production of sulfur concrete requires and use of sulfur high technological expertise, and as such, the use of sulfur concrete has only been limited to special applications. The production of such sulfur concrete is such that the sulfur is heated up to be re-active and used as a complete replacement of cement. Hence, the sulfur here acts as a binder.

This research is an exploratory experiment into sulfur concrete used not as a complete replacement of cement but as an additional material in percentage of the cement content. The aim of this research was to explore the possible appreciation of mechanical and physical properties of concrete containing sulfur with percentages of 1%, 5% and 10% of the cement content. The sulfur used here was not heat-activated, hence the binding effect in sulfur was absent. The experimental results revealed that concrete containing sulfur did not perform better in their strength properties, both compressive strength and flexural strength. The physical properties such as water penetration and water absorption for concrete containing sulfur also showed poor performance in comparison to ordinary Portland cement concrete. Such phenomena are very likely due to the sulfur not being activated by heat. Carbonation test did not show good results as a longer term of testing is required. Drying shrinkage property was found to be encouraging in that concrete containing 10% sulfur had quite significant reduction in drying shrinkage as opposed to ordinary Portland cement concrete. Further research of concrete containing sulfur that is heated up during concrete mixing will be useful to draw better conclusions. Research to measure corrosion resistance in sulfur concrete, for instance using half-cell potential approach will be an interesting finding and contribution.

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