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Research

Spent coffee grounds enhanced compressive strength of cement mortar: an optimization study

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Abstract

This paper presents an optimization study of spent coffee grounds (SCG) as cement mortar additives to enhance mortar strength. In recent years, sustainable materials have begun finding their way into cement mortar, with SCG being one. There is limited optimization study on the SCG addition in mortars, hence this study was performed to optimize the curing time and SCG addition in cement mortar to achieve the highest compressive strength through response surface methodology. Scanning Electron Microscopy (SEM) characterization was carried out on the SCG particles to identify their physical properties. An Energy Dispersive X-ray (EDX) analysis was carried out to identify its chemical properties. Simultaneously, a workability test, the flow table test, is conducted to study the effect of SCG on the flowability of the cement mortar mixes. The synergistic effect between SCG content in cement mortar mixes and the curing period was statistically studied and analyzed. Both parameters were then optimized to obtain the best performance mix of SCG in cement mortar. It was found that 1.1% SCG and a curing day of 68 days produced the highest compressive strength (33.4MPa) of cement mortar. The Response Surface Methodology (RSM)-optimized cement mortar mix presented at least a 12.62% improvement in compressive strength from control cement mortar without SCG additives (28.77MPa). Experimental validation of the optimum condition showed a good agreement with a deviation of 3.12% in three replicates, thus indicating that the optimum model in this work can be used to model the compressive strength of the SCG-cement mortar mixture.

Article Highlights

1. The addition of spent coffee grounds (SCG) in cement mortar enhanced its strength by 12.6%.
2. The optimum SCG addition that corresponds to the highest mortar strength is 1.1%.
3. The flowability of cement mortar mix decreases as the percentage of SCG addition increases.

Keywords Response surface modelling · Additives · Spent coffee grounds · Cement mortar · Optimization · Characterization

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1 Introduction

Concrete is a type of man-made composite material made with the combination of a cementitious binder and aggregates. Concrete, in its fresh state, is flowable and acts like a paste. However, upon setting and hardening, the concrete starts to gain strength and become tough. For that, concrete has been widely applied in the construction industry today, for the construction of roads, houses, skyscrapers, and many more [1]. Due to the wide range of applications of concrete, there is a rising trend in the use of additive materials in concrete to induce special behaviour in the concrete mix. Some types of additive materials come in the form of a liquid solution while others come in the form of powder [2, 3]. Additive materials have a variety of influences on concrete behaviour. Certain additives could slow down the rate of setting of a concrete mix, increase the workability of a concrete mix, or even improve the strength of concrete over the long run [4, 5]. With concrete being named the second most used substance in the world, the detrimental effects of concrete on the environment should not be ignored [6, 7]. Therefore, many studies have tried to increase the service life of concrete in hopes of reducing the consumption rate of natural minerals needed to make concrete, and also to use waste materials that are of no commercial value into concrete to improve the properties of concrete [8–11].

In recent years, there has been a growing number of research being conducted on the utilization of wastes in cement mortar mixes. Based on empirical and quantitative research, many have scientifically recorded improvements in concrete properties when mixed with sustainable materials [12, 13]. Though there has been considerable research conducted in the field of reutilization of wastes as sustainable materials, the concept of sustainable cement mortar has not been largely popularized in the construction industry [14]. Previous research has identified that among the many reasons, one main concern of the industry is the accessibility of the waste materials that could be reutilized into concrete [15, 16]. With coffee drinking being a common practice around the world today, spent coffee grounds (SCG) become a commonly available waste that could be obtained from any shop serving coffee beverages or even at any household that brews coffee [17]. Waste materials such as palm oil fuel ash and rice husk ash were found to improve concrete properties, however accessibility of these wastes in extremely cold or hot countries is scarce, but SCG could be found just about anywhere [18, 19]. Therefore, this work aims to find the optimum content of SCG in a cement mortar mix that corresponds to the highest gain in compressive strength of cement mortar.

SCG are wastes obtained from the process of coffee brewing. To make coffee, roasted coffee beans are first ground into finer sizes. The fineness of the coffee grounds is based on the coffee-drinking culture of the locality. In Malaysia, the coffee-drinking culture in traditional coffee shops is grounded in a medium size. To make the drink, hot water is poured over the coffee grounds and sieved over a cloth strainer. The method is almost similar to pouring over coffee of the coffee-drinking culture except it is done manually. When the coffee grounds ran out of flavour, the leftovers on the strainer, also known as spent coffee grounds (SCG) were discarded as general wastes.

Coffee consumption in Malaysia for the year 2022 is about 48,000 tonnes [20]. The spent coffee represents 65% of the coffee beans' lifecycle, thus roughly 31,200 tonnes of coffee would eventually end up as spent coffee thrown into landfills if no reuse effort is on the way [21]. On the global scale, the International Coffee Organization reported that coffee consumption in the year 2022 alone amounts to 10.54 million tonnes [22]. These SCGs, seemingly harmless, are threatening to the environment when left to decay in landfills. SCG leachate formed during decomposition is known to be acidic and forms greenhouse gases [23]. At large volumes, this waste could potentially result in infertility of soils and harmful air contamination [24]. For that, a rising body of literature has begun examining the possibility of revitalizing SCG wastes, but there is a limited study on the optimization of SCG content in cement mortar mixtures [25–27].

There have been several studies on SCG used in cement mortar mixes as additives in the past. In a study utilizing SCG as additives in cement mortar, Chua [28] reported that the compressive strength of the cement mortar increases, but the involvement of SCG as part of the mix design is only at 0.6%. Another study by Shahdin et al. [29] reported that when using SCG as an additive in cement mortar up to 4%, all percentages of the incorporation show a drop in strength. From previous literature, while substantial research has been conducted on the use of SCG in cement mortar, it is evident that there are no numerical and statistical models yet that could establish a cement mortar recipe for mortars containing SCG. Thus, the main aim of the current work is to investigate the properties of cement mortar containing SCG at different percentages. The percentages of SCG and curing times are analysed using response surface methodology to formulate the best recipe for SCG cement mortar mixes to achieve the highest compressive strength.

From previous literature, the SCG used as additives in cement mortar were mainly sourced from Western coffee shops. The SCG in this study, however, is sourced from traditional Malaysian coffee shops. While both are universally known as SCG, the difference in sources of SCG wastes could lead to differences in its properties. This mainly stems from the different roasting methods for the coffee beans as well as the different genotypes of coffee beans [30]. Western coffee shops typically use different fermentation and roasting processes for the coffee beans to make their coffee [31]. This is different from the Malaysian traditional coffee culture where the coffee beans are usually roasted with sugar, similar to a roasting method known as the Torrefacto method [32]. This minimal difference in sources could lead to a difference in strength performance. This can be seen from previous studies that when utilizing olive waste and wood biomass ash, differences in sources lead to differences in basic properties of the biomass ash and the end affecting the strength of the concrete [33]. Unfortunately, no studies are known today yet in using SCG originating from traditional Malaysian coffee shops.

It is well established by previous research that cement powder present in mortars requires water for the hydration to take place in the cement mortar matrix to form calcium silicate hydrate (CSH) gel [34]. CSH gel acts like a binder in a cement mortar matrix to hold the mortar constituents together. At fresh state, when mixed with water, this cement powder becomes a gel-like paste and upon setting and hardening, the gel then solidifies to form a strong bond between aggregates of the concrete, giving the concrete its strength [35]. This phenomenon is explained to take place as a result of more cement particles becoming hydrated over the days of curing in water. However, the rate of strength gain in concrete tends to show a high increment over the first 28 days and slows down after 28 days of water curing [36]. Nevertheless, when concrete mixes were blended with sustainable materials, multiple previous studies have identified that the strength gain could be observed even up to 365 days of water curing [37]. While previous studies have identified the effect of SCG on cement mortar strength up to 28 days, the effect of water curing at extended periods after 28 days has not yet been established. Therefore, for this study, a water curing of a maximum of 70 days was adopted to identify the effect of SCG in providing strength to the cement mortar mix after 28 days of water curing.

To further identify the effects of curing time and SCG content on the compressive strength of cement mortar mixes, Response Surface Methodology (RSM) is adopted. RSM is a mathematical and statistical method to study the relationships between multiple manipulated variables to one or more response variables. The main purpose of conducting RSM modelling is to properly analyse experimental data and establish a general understanding of a particular field of interest. Setting both the curing time and SCG content in the cement mortar mixes as the manipulated variables, the compressive strength performance of the cement mortar is evaluated. To investigate the validity of the findings, Analysis of Variance (ANOVA) was adopted to identify the error and significance of the experimental data set.

Ultimately, this study aims to identify the potential of SCG as an additive in improving the performance of cement mortar. To initiate the study, SCG is being characterized through Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray (EDX) techniques to obtain a foothold on its physical and chemical properties. To dive further into understanding the potential of SCG as construction materials, as a preliminary investigation, this study utilizes cement mortar mixes containing SCG as an additive. The workability and mechanical performance of cement mortar containing SCG additives are quantitatively studied. Then, Response Surface Methodology (RSM) optimization is carried out to study the most optimum level of SCG additive to produce a cement mortar with high strength. Ultimately, this study establishes a substantial knowledge of the capacity of SCG as an additive in cement mortar mixes paving the way for future works in studying its application in mortar mixes.

2 Materials and methods

2.1 Spent coffee grounds (SCG) preparation

The spent coffee wastes were collected from Pusat Kedai Makanan Odawala, a coffee shop situated in Bagan Serai, Perak, Malaysia. At first, the coffee waste was washed with water to remove residual coffee liquid. Subsequently, the coffee waste was dried in an oven at 110 ± 5 °C until the temperature reached constant mass, indicating a complete moisture removal. The coffee waste was then cooled to room temperature and then ground using a Los Angeles Abrasion Machine whereby 21 metal balls were inserted into the chamber together with the dried coffee wastes for crushing in 4500 cycles. The grounded coffee waste (SCG) in powder form was sieved through a 45-micron sieve before being used as an additive in cement mortar. The sieved SCG powder was stored in a plastic container in a cool dry place containing silica gel desiccant to prevent moisture absorption until further use.

The processed spent coffee powder is then sent to Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray (EDX) techniques for characterization. SEM technique is a method of morphological investigation that largely magnifies the image of a material using electrons. For this study, SCG is magnified up to 500 times and 1000 times to investigate its physical properties under high vacuum. EDX analysis is a method to identify the chemical composition of a material through the use of X-ray beams. The technical configuration for the EDX analysis is shown in Table 1. The SEM–EDX investigation in this study is conducted through the use of the JEOL JSM-IT200.

2.2 Cement mortar preparation

The cement mortar mixes were prepared using a cement-to-sand ratio of 1:3 and a water-cement (w/c) ratio of 0.5. The effect of SCG addition in cement mortar was studied by adding the SCG at various percentages up to 3%. According to Chua [28], SCG additives in cement mortar could only be effective up to 0.6%. However, a study by Shahid et al. [29] showed that when SCG was added as an additive in cement mortar, the compressive strength of the cement mortar generally decreased, with extremely low strength data reported from 1% additive up until the 4% mark. The finding from the aforementioned work is tinged with confusion and no optimization study was performed. Thus, the SCG addition in cement mortar ranging from 1 to 3% was selected for optimization in this work.

The cement utilized in this study is composite Portland cement (PCC) supplied by YTL Cement Limited under the product name CASTLE which is certified according to Malaysian Standard MS EN 197–1: 2014, Portland limestone cement, CEM II /B-L 32.5N. The PCC used in this work is of Grade 33 with a normal strength gain class. The PCC used in this study is also a mixture of ordinary Portland cement (OPC) blended with a mixture of limestone at a range of 21 to 35%. The technical specifications and chemical properties of the Portland composite cement used in this study are shown in Table 2. The cement-to-sand ratio of 1:3 was used which is analog to the Type-N cement mortar according to the ASTM C1329-05 standards [38].

Table 1 Technical configuration settings to investigate the SCG particles in this study

Items	Value
Measurement conditions	
Acceleration voltage	10.00 kV
Magnification	X 1000
Process time	T3
Live time	30.00 s
Real time	30.17 s
Dead time	1.00%
Count rate	255.00 CPS
Quantification	ZAF
Fitting ratio	0.0783

Table 2 Technical specifications of Portland composite cement used for this study

Tests	Units	Test Results
Chemical composition		
Sulfate content (SO_3)	%	2.1
Chloride (Cl)	%	0.01
Physical properties		
Fineness	m^2/kg	440
Setting time (initial)	Minutes	155
Soundness	mm	0.8

Table 3 Mix design of cement mortar containing Spent Coffee Grounds (SCG) additives deployed for this study

	Sand (kg/m^3)	Cement (kg/m^3)	SCG (kg/m^3)	Water (kg/m^3)
SCG 0%	3000	1000	0	500
SCG 1%	3000	1000	10	500
SCG 3%	3000	1000	30	500

Table 4 Experimental design deployed for the study

Design Points	x_1– Curing Period (Days)	x_2– SCG Content (%)	y– Compressive Strength (MPa)
1	7	0	19.07
2	7	1	20.95
3	7	3	6.25
4	28	0	24.23
5	28	1	28.23
6	28	3	15.28
7	70	0	28.77
8	70	1	34.36
9	70	3	20.74
10	7	0	19.07
11	70	0	28.77
12	70	3	20.74
13	28	1	28.23

Table 5 ANOVA for Central Composite Design of the quadratic model

Source	Sum of squares	Degrees of freedom	Mean square	F-value	p-value
Model	632.06	5	126.41	231.1	1.30×10^{-7}
x_1–curing period	318.54	1	318.54	582.34	5.30×10^{-8}
x_2–SCG content	213.14	1	213.14	389.65	2.10×10^{-7}
x_1x_2	7.06	1	7.06	12.91	0.0088
x_1^2	19.71	1	19.71	36.04	0.0005
x_2^2	124.32	1	124.32	227.28	1.40×10^{-6}
Residual	3.83	7	0.547		
Lack of fit	3.83	3	1.28		
Pure error	0	4	0		
Cor Total	635.89	12			

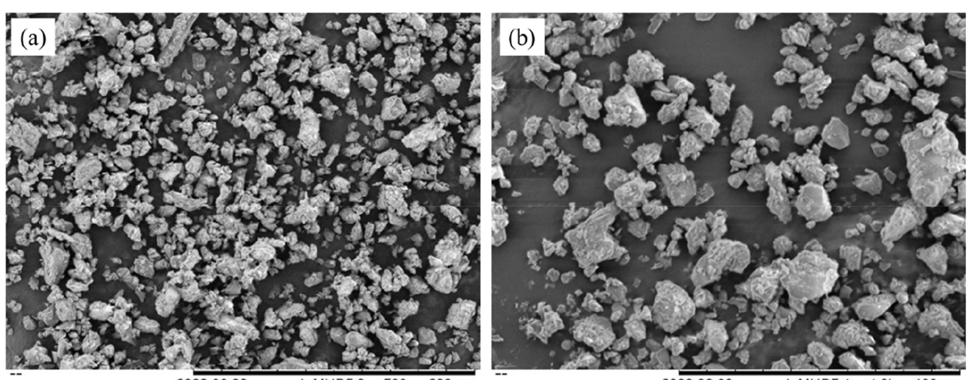

Fig. 1 SEM image of SCG particles at different magnifications. **a** 500×, **b** 1000×

The mix design deployed for the study is shown in Table 3. The sample was assessed for their workability, and mechanical performance. Workability is a crucial factor in the strength performance of a cement mortar. This was observed previously by Yogesh et al. [39], who reported that the workability of the cement mortar has a high positive correlation to its strength. It is known that high workability may cause a reduced strength of the cement mortar. Meanwhile, low workability is associated with high mortar strength according to Raju et al. [40]. However, it is important to note that the low workability of mortar mix makes it hard to place and handle, which could lead to cracking issues. Thus, it is important to identify the most optimal workability of a mix that could form cement mortar with improved strength.

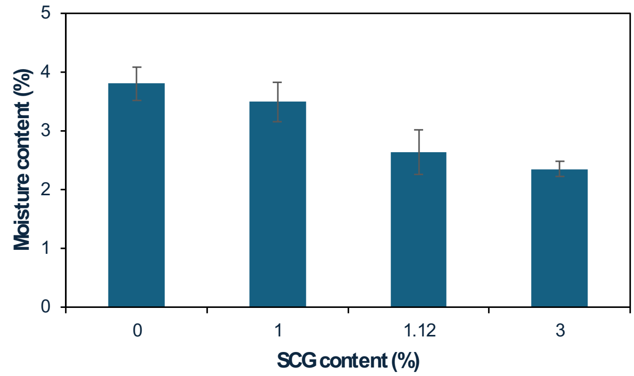

Fig. 2 Water absorption of cured cement mortar containing SCG from 0 to 3%

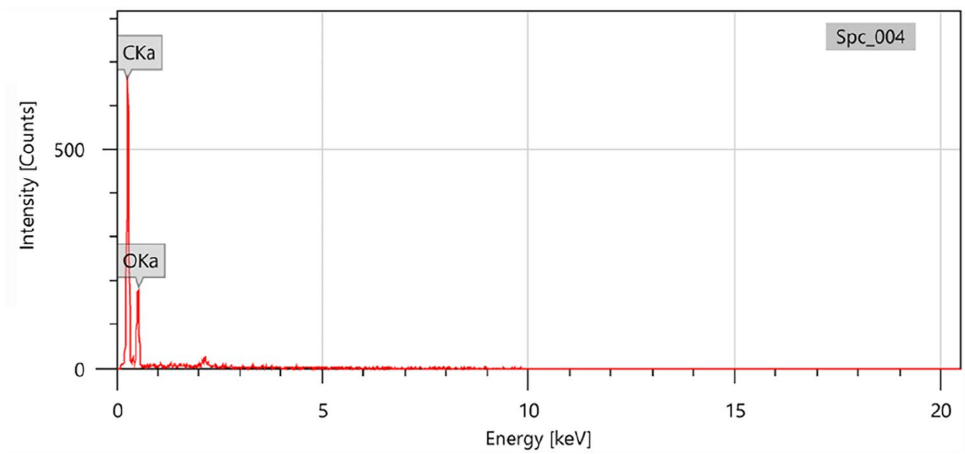

Fig. 3 Chemical characterisation of the SCG through Energy Dispersive X-Ray (EDX) analysis spectrum

The workability is evaluated through a flow table test, while the strength performance is investigated through a compressive strength test. The flow table test of cement mortar mix is tested when mortar mixes are still fresh and workable according to ASTM C1437 standards [41]. The flowability of the cement mortar mixes is calculated according to Eq. (1), as a function of the average four mortar diameter readings, A, and original base diameter, B. After the flow table test, the fresh mixes were then put into a mortar cube measuring 50 mm × 50 mm × 50 mm. The fresh mixes were placed in two batches into the mould, with each batch subjected to vibration on the vibrating table for compaction. Subsequently, the mixes were allowed to be set for 24 h on a drying rack before being removed from the mould for curing with water immersion. For each percentage of SCG additive, 3 cube samples were tested for their compressive strength during a water curing period of 7-day, 28-day, and 70-day. Conventionally, the compressive strength of cement mortar at 28 days is taken as the ultimate strength performance. This is due to the cement known to have fully set and gained its maximum strength at 28 days. However, for this study, extended curing periods of up to 70 days were selected as previous literature has identified that improved strength could be observed in cement mortar containing SCG additives when cured at extended periods of over 28 days. From the 3 samples, the average compressive strength value will then be taken to ensure for reproducibility of the data. The compressive strength test of the cement mortar was conducted according to ASTM C109/C109M-20 standards [42]. Water absorption of the mortar sample containing SCG ranging from 0 to 3% in this work was measured using a thermogravimetric analysis (STA7000, Hitachi). All samples were placed in a pan and heated from 20 °C to 800 °C at a heating rate of 10 °C/min under a nitrogen atmosphere. Heat loss recorded from 80 to 250 °C, was taken as heat loss associated to loss of moisture from the specimen.

$$Flowability\ (\%) = \frac{A - B}{B} \times 100\%, \tag{1}$$

Table 6 Energy Dispersive X-Ray (EDX) analysis of the SCG particles at a fitting ratio of 0.0783

Element	Line	Mass (%)	Atom (%)
C	K	66.85 ± 0.59	72.87 ± 0.64
O	K	33.15 ± 1.00	27.13 ± 0.82
Total		100.00	100.00

2.3 Optimization study

The response surface methodology was performed according to a central composite design. The compressive strength of the cement mortar was used as a response, while the main are the curing days (x_1) and the content of SCG additives in the cement mortar mixes (x_2). The curing days (x_1) were set to have a range of a minimum of 7 days to a maximum range of 70 days as the maximum. Meanwhile, for the content of SCG additives in the cement mortar mixes (x_2), the lowest level was set as 0% whereas 3% is set as the maximum of the design. The values were set to be from -1 to 1 for both parameters. The response parameter for the optimization study is the compressive strength (y) with MPa as the unit. To run the optimization model, a total of 13 experimental runs were required. The model requirements of design points and the experimental values of all 13 experimental runs are shown in Table 4. ANOVA was performed to examine the appropriateness of the data (Table 5). After the 13 experimental runs were conducted, the analysis of the design points obtained from the central composite design (CCD) was then plotted into a response surface methodology plot (RSM). The RSM plot is fitted to the second-order polynomial equation with the design points from the CCD analysis. The model showed an insignificant lack of fit and the model is indicated as significant. The $R^2 = 0.99$ was obtained indicating excellent fitting between the model equation and the experimental data (Table 4). The highest compressive strength was selected as a desired solution with both factors x_1 and x_2 kept in range. Experimental validation for the optimum condition obtained from RSM was performed to ensure the validity of the model.

3 Results and discussion

3.1 Characterization of spent coffee grounds (SCG)

The surface morphology of SCG obtained from the SEM is presented in Fig. 1. It could be observed that SCGs that have been ground and sieved in this study are irregularly shaped. Compared to previous studies on the particle characteristics of cement, it can be said that SCG has the same irregular shapes. These irregular shapes of the cement, which are present in SCG, are said to be beneficial for the cement mortar mixes as they help increase the interlocking faces of the mortar matrix [43]. Many studies have been conducted on fly ashes and the ability of fly ashes to improve cement mortar strength is well established. However, under SEM imaging, previous studies have reported that fly ashes are materials with spherical particles, and that differs from that of the SCG particles [44]. Other than the irregular shapes of the SCG particles, another important characteristic found present in the SCG particles was its high susceptibility to moisture. This meant that the SCG posed higher water absorption properties. Earlier, Lee et al. [45] studied in detail the effect of water absorption by SCG in the cement mix. They found that although SCG

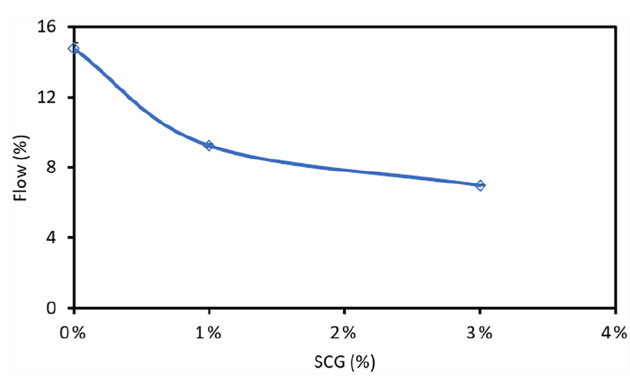

Fig. 4 Flowability of the SCG cement mortar obtained through the flow table test

absorbs water, however, the detrimental effect is only significant at 6% SCG content. Lee et al. [45] also reported that the addition of SCG increased the compressive strength of cement mortar with the highest strength attained with 0.9% SCG addition. Nevertheless, the mix containing 1.8% SCG also still somewhat produced improved compressive strength of the cement mortar. Since Lee et al. [45] did not perform an optimization study, it is difficult to pinpoint exactly where the optimum SCG content should be, and the aforesaid issue was overcome in this work.

The water absorption capacity of SCG is about 70% wt. which was measured by heating the saturated SCG until a constant mass is attained at 110 ± 5 ºC. However, it should be noted that the mortar preparation in this work makes use of the dried SCG. The moisture content in cured mortar containing SCG ranging from 0 to 3% which covers all the range studied in this work was performed using a thermogravimetric analysis. The results in Fig. 2 show that moisture content in cement mortar decreases as SCG content increases to 3%. The moisture content in a hardened mortar containing 3% of SCG is lower than that of the control sample. This may be attributed to the hydration process of concrete. When cement comes in contact with water, water is consumed for a hydration reaction converting cement powder into calcium silicate hydrate gel, a gel-like liquid which upon hardening bonds the cement mortar constituents together [46–48]. Hence, at its fresh state, when SCG is added into the cement mortar mixes at 1%, excess water in the matrix could be absorbed, and hardening results with SCG present in the matrix to fill in the internal voids of the cement mortar leading to decreased water absorption capacity. At a 3% addition of SCG, at its fresh state, the SCG would have ended up absorbing most of the water that is required of the cement to undergo a hydration reaction resulting in lowered water content in the matrix. Though upon hardening, the SCG could fill in the voids of the cement mortar the incomplete hydration reaction of the cement powder resulted in stunted C–S–H gel formation, which is crucial in strength development. Previous work by Lee et al. [45], also shows similar findings on reducing moisture content for cement mortar containing SCG up to 3%. Although, a higher SCG content (6%) water absorption increases markedly to about 5% wt. From our preliminary screening, it was found that no gain in concrete strength at higher SCG content beyond 3%.

The chemical properties of the SCG obtained from the EDX are presented in Fig. 3. The intensity peak of the elements is tabulated as shown in Table 6. From the EDX analysis, carbon has the highest intensity in SCG with 66.85% of the total mass. The second highest composition is oxygen with an intensity peak of 33.15%. Carbon makes up 72.87% of atomic percentage while oxygen takes up 27.13%. Apart from these two elements, no other chemical elements were observable in the SCG particles. The results show that SCG used in this study could not be classified as pozzolanic materials as it does not have any pozzolanic properties satisfying the ASTM C618, C989, and C1240 standards [49–51]. Identifying the high carbon content in the SCG indicates that SCG could not be utilized at high percentages in a concrete or cement mortar mix. Carbon is not reactive and does not react with water under normal conditions [52]. This makes carbon take up space in the cement mortar matrix, reducing the volume of CSH gel that contributes to the strength gain of the cement mortar. Thus, this limits the amount of SCG that can be added to the cement mortar and concrete. This can be observed through previous literature which has also reported that carbon black, a material high in carbon content (85%) could not be added at a high percentage into the cement mortar. At percentages higher than 10%, the strength dropped drastically [53].

3.2 Workability of spent coffee grounds (SCG) cement mortar

The workability of cement mortar when SCG was mixed as an additive material is shown in Fig. 4. From the flow table test, it can be observed that at increasing additive proportions in the cement mortar, the workability of the mix decreases. This is due to the inherent properties of SCG which is susceptible to moisture, hence more water was absorbed by SCG

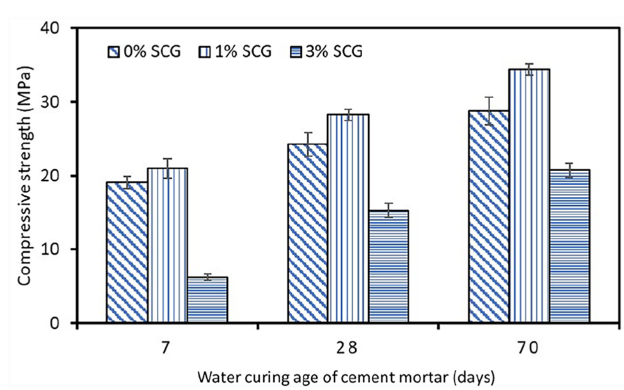

Fig. 5 Compressive strength of the SCG cement mortar at different water curing periods

as its content increased in the mix. This led to a decrease in the flowability of the cement mortar when the SCG increased from 0 to 3%.

According to Nasir et al. [54], the decrease in flowability of the cement mortar mixes could mean an increase in its strength. Sabrin et al. [55] also reported a similar finding whereby the increase in flowability decreases the ultimate strength of the cement mortar. Nevertheless, too high or low workability may lead to a decrease in compressive strength. Thiedeitz et al. [56] for instance reported that increasing rice husk ash (RHA) content was found to decrease the workability and compressive strength of cement mortar. This could be due to the reaction kinetics of cement in the concrete matrix. The hydration reaction of cement with water is complex. However, it mainly involves Tricalcium Silicate (C_3S), Dicalcium Silicate (C_2S), Tricalcium Aluminate (C_3A), and Tetracalcium Aluminoferrite (C_4AF) compounds [57]. Looking into the Tricalcium Silicate (C_3S) reaction, the equation is shown in Eq. (2) [47]. From the reaction equation, it can be concluded that the cement relies heavily on water to form a calcium silicate hydrate compound. These compounds are gel-like substances when they are fresh, but when allowed to set and cure, these compounds harden, becoming the main contributor to the strength gain of concrete.

In the case of SCG, these SCG could adversely affect the cement mortar strength, as much of the moisture was absorbed by the SCG, leaving only a low content of water in the cement mortar matrix for the continuous hydration reaction to take place and form more calcium silicate hydrate gel. Instead, these SCG absorb a high amount of water in the cement mortar matrix, leading to a low flowability but decreasing strength phenomenon. This is observed when the cement in a mix fails to be hydrated properly into a gel, this cement powder would harden prematurely, leaving high amounts of unreacted cement powder in the mortar mix, thus leading to the lower strength phenomenon.

Comparing the results obtained from previous studies conducted on SCG cement mortar, Chua [28] reported that the increasing amount of SCG incorporated from 0.2% to 0.8%, the lower the flowability. However, there is an increase in flowability towards the 1% SCG additive. Another study by Shahid et al. indicated similar flowability values where the flowability decreases when incorporated up to 0.75% and increases towards 4% additive. For this current study, however, the flowability did not increase but decreased throughout all 3 different ranges of percentages. This could be due to the hygroscopic property of the SCG which led to water in the cement mortar mix being absorbed, resulting in low water content present in the mortar mix, leading to the drop in flowability. The differences in the results between the current study and previous literature could also be due to the difference in sources of the SCG materials, where both Chua [28] and Shahid et al. [29] have reported using SCG obtained from western coffee shops while this study utilizes SCG obtained from traditional Malaysian coffee shops. The differences in sources could lead to the differences in the properties of the SCG wastes as different coffee cultures have different methods of processing the coffee beans to make coffee.

$$C_3S + (3 - x - n)H_2O \rightarrow C_x - S - H_n + (3 - x)CH, \tag{2}$$

3.3 Compressive strength regression model of SCG cement mortar

After obtaining the flowability values of the SCG cement mortar, the fresh mixes were cast into mortar cube molds measuring 50 mm on each side. The SCG cement mortar is then allowed to harden and cure for over 70 days. The compressive strength of the SCG cement mortar is measured on 7 days, 28 days, and 70 days of water curing. The compressive strength result is shown in Fig. 5. From the results, it can be observed that the highest compressive strength was found in cement mortar mixes containing 1% SCG. The lowest-performing cement mortar mixes are those containing 3% SCG content. Throughout the 70 days of curing, the cement mortar mixes showed continuous strength gain regardless of the content of SCG in the mix. Thus, these findings agree with the earlier studies reported on the workability of the cement mortar mixes where decreasing workability does not necessarily imply increasing compressive strength as

Table 7 Fit statistics of the regressed model

Fit Statistics			
Standard deviation	0.74	R^2	0.99
Mean	22.67	Adjusted R^2	0.99
Coefficient of variation %	3.26	Predicted R^2	0.98
		Adequate precision	53.24

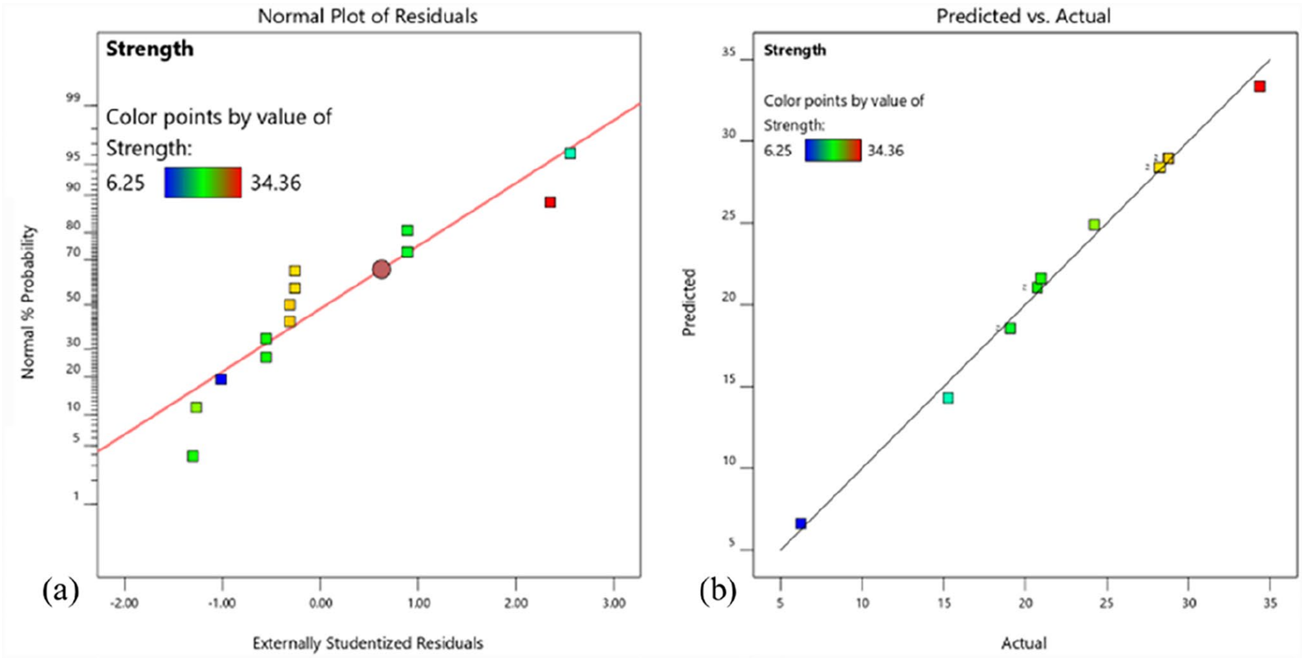

Fig. 6 **a** Normal plot of residuals. **b** Predicted vs. experimental data plot

water needed by the cement hydration process is limited due to the presence of SCG, which is a material that has high water absorption tendency.

With the addition of 1% SCG in the cement mortar mix, the strength increases and outperforms the control mix. This could be due to the SCG, a hygroscopic material being able to absorb excess water in the cement mortar mix. When cement comes in contact with water, a cement hydration process takes place inside the mortar matrix. However, after the hydration phase, minor shrinkage could take place and is known to form around 8% of micropores in the concrete [58]. This situation is worsened when excess water that may be present in the mortar has been left unreacted in the mix [59]. Drying this unreacted water would eventually result in increased tiny air pockets forming inside the mortar matrix [60]. Thus, the presence of SCG could help to absorb this excess water in the mortar matrix and allow for a more compacted inner matrix as cement particles could continue to flow into these pores before hardening. The unreactive SCG due to its high carbon content could also allow for the SCG to not have any adverse reaction within the matrix but act like a filler additive in improving the compactness of the cement mortar [61].

However, at higher quantities up to 3%, the presence of SCG is too high. The SCG could end up occupying too much volume in the cement mortar matrix. Unreacted, these SCG would not contribute to strength gain, but adversely increase the amount of micropores of the cement mortar matrix, leading to a decrease in compressive strength [62]. High quantities of hygroscopic spent coffee grounds in the cement mortar mix could also lead to cement hydration not being able to take place due to the lack of water particles to hydrate the cement particles [63]. Water is crucial for the formation of C–S–H gel, this could be seen in Eq. (2). Instead of encouraging cement hydration, the SCG particles may end up competing for water particles in the cement mortar matrix, leading to incomplete cement hydration.

In previous works on cement mortar containing SCG additives, Chua [28] reported that at 28 days of curing, cement mortar without SCG presented the highest compressive strength, however, at extended curing period of up to 45 days the cement mortar with the highest compressive strength could be observed in 0.6% SCG additives with 0.4% SCG and 1.0% SCG having improved performance in compared to the control cement mortar. The findings of this study differ as cement mortar containing 1% SCG additives have been able to outperform the control mixes from a curing age of 7 days and the same trend is observed up to 70 days of curing. On the other hand, the findings of the study show a difference in strength performance compared to Shahid et al. [29], where even at an extended period of curing, SCG once added into the cement mortar shows detrimental strength values with no SCG cement mortar outperforming control mixes.

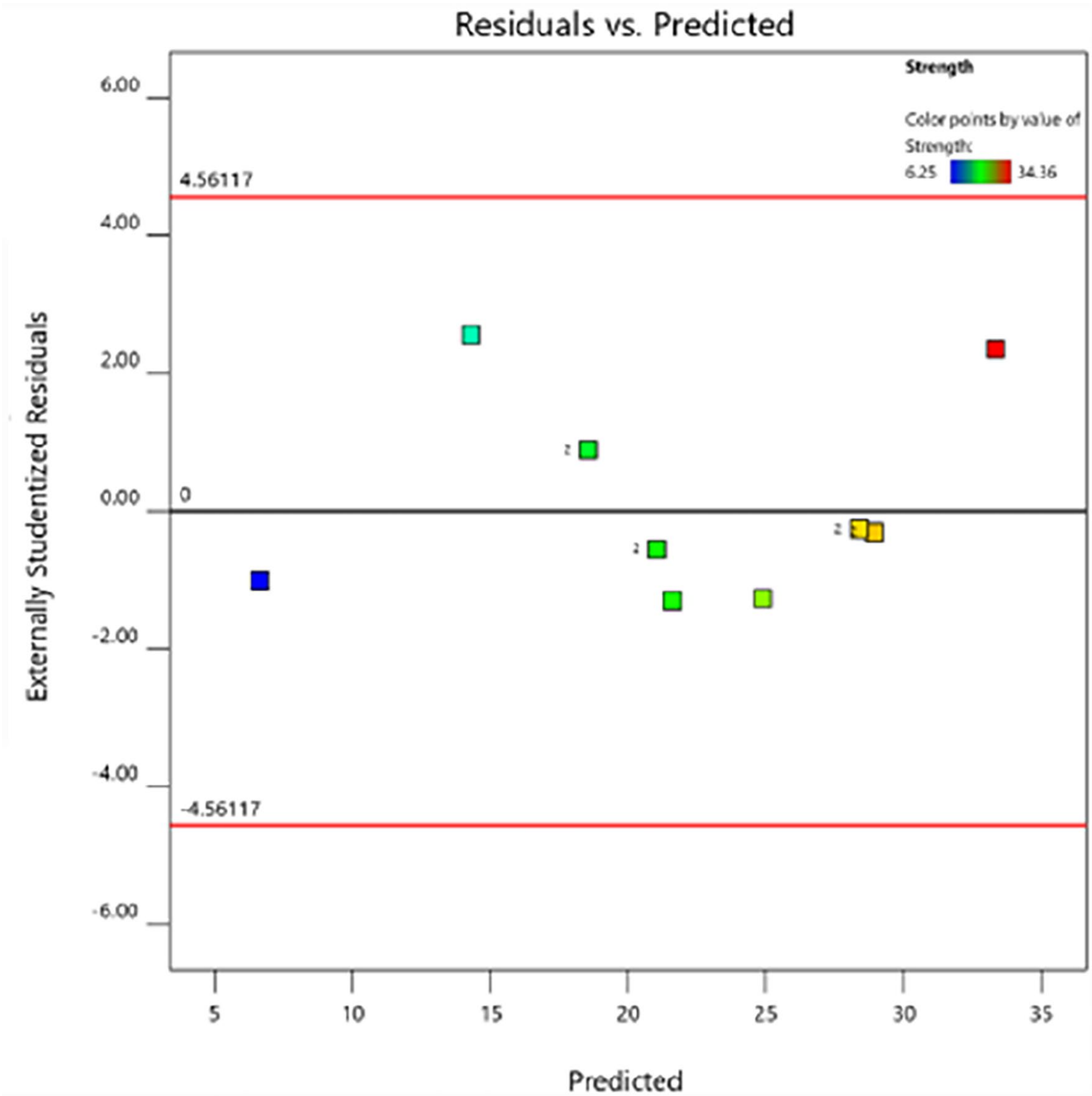

Fig. 7 Residual data against predicted data

As mentioned earlier in Sect. 3.2, the SCG used in this work differs from that of Chua [28] and Shahid et al. [29], which could be attributed to the difference in the findings.

A regression model was used to understand the interrelation between the compressive strength of the SCG cement mortar with the curing period and SCG content. A quadratic model was used for the RSM due to a good fit as seen from the ANOVA result. The ANOVA result is shown in Table 5. It can be seen that the model contains an F-value of 231.10. The high F-value indicated that this CCD model significantly represented the data with minimal error (0.01%). The p-values of all the individual terms, Curing Period (x_1) has a p-value of 5.3×10^{-8} and SCG content (x_2) has a p-value of 2.1×10^{-7}, whilst $x_1 x_2$ has 8.8×10^{-3}, x_1^2 with 5×10^{-4} and x_2^2 with 1.4×10^{-6}. All these p-values could be seen to have values lesser than 0.05, indicating that all these parameters are significant model terms that have to be included in the model. From the analysis, the model was able to form a quadratic equation as shown in Eq. (3). The $R^2 = 0.99$ implies that there is a strong positive correlation between the quadratic model and the experimental data. Between the model and experimental data, the standard deviation value is low (0.74). The coefficient of variation (CV) value is 3.26%, hence the regressed model can predict the experimental values accurately. The difference between the model R^2 and adjusted R^2 is less than 0.2, showing that the model is acceptable without significant adjustments (Table 7). The adequate precision value is also 53.24 (> > 4), thus the model is valid and sufficient to navigate the design space.

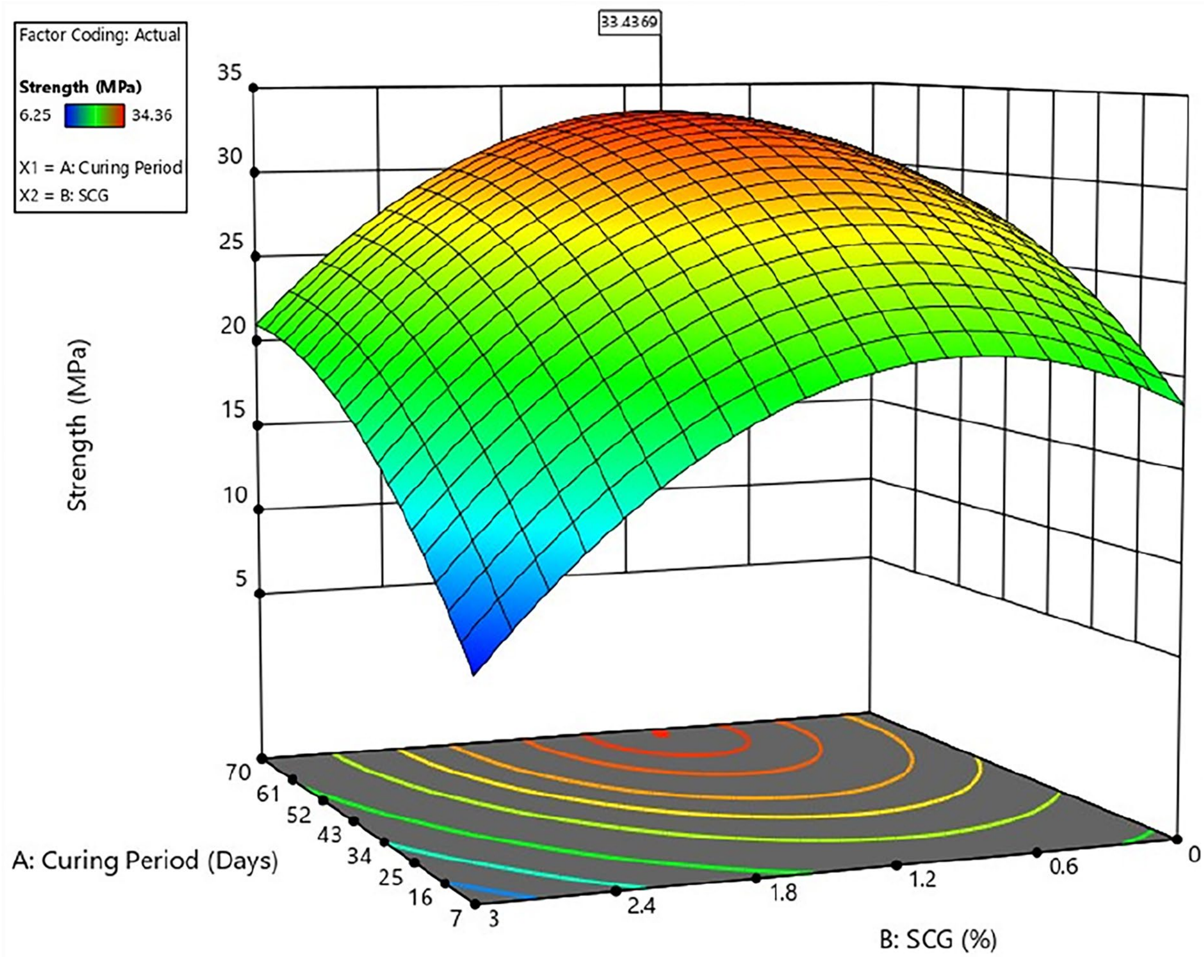

Fig. 8 3D response surface plot of SCG content and curing days to strength of cement mortar

Figure 6 shows the graph for the normal plot of residuals and predicted vs. experimental value. It can be seen that the predicted value is in good agreement with the experimental data. From the Box-Cox plot for power transforms, the best lambda value obtained is 0.96 while the current lambda used for the optimization study is 1. Box-Cox transformation is a statistical method of transformation of non-normal experimental data into a normal distribution. The transformation is to improve the accuracy of the prediction of the experimental data. In a Box-Cox transformation, the lambda value represents the confidence interval of the model to represent the original data. If the lambda value is close to unity means that the predicted data is equivalent to the experimental data, hence no transformation is needed.

A graph of residuals against predicted is plotted as shown in Fig. 7. It can be seen that the random distribution of experimental data points lies well within the acceptable range. The values of all the randomly distributed data do not exit the acceptable threshold. In terms of the Cook's Distance plot of the data, all the data lies well within the thresholds. This meant that there were no outliers present in the data and all data of the experimental work were normal. The model equation is generated as shown in Eq. (3) with the regression model obtained and validated. The second-order polynomial quadratic model is then plotted into a three-dimensional response surface graph (Fig. 8). The interaction between the compressive strength of the SCG cement mortar, curing periods, and SCG content in the mix is then investigated through the interaction plot shown in Fig. 9 and the perturbation plot in Fig. 10.

$$y = 29.94 + 6.21x_1 - 4.96x_2 + 1.01x_1x_2 - 3.22Ax_1^2 - 7.92x_2^2, \tag{3}$$

The relation between the curing period and SCG content in the cement mortar with the compressive strength is observed from the 3D response surface plot. It can be seen that the compressive strength of the SCG cement mortar increases with the SCG content in the cement mortar up to around 1% before experiencing a steady drop

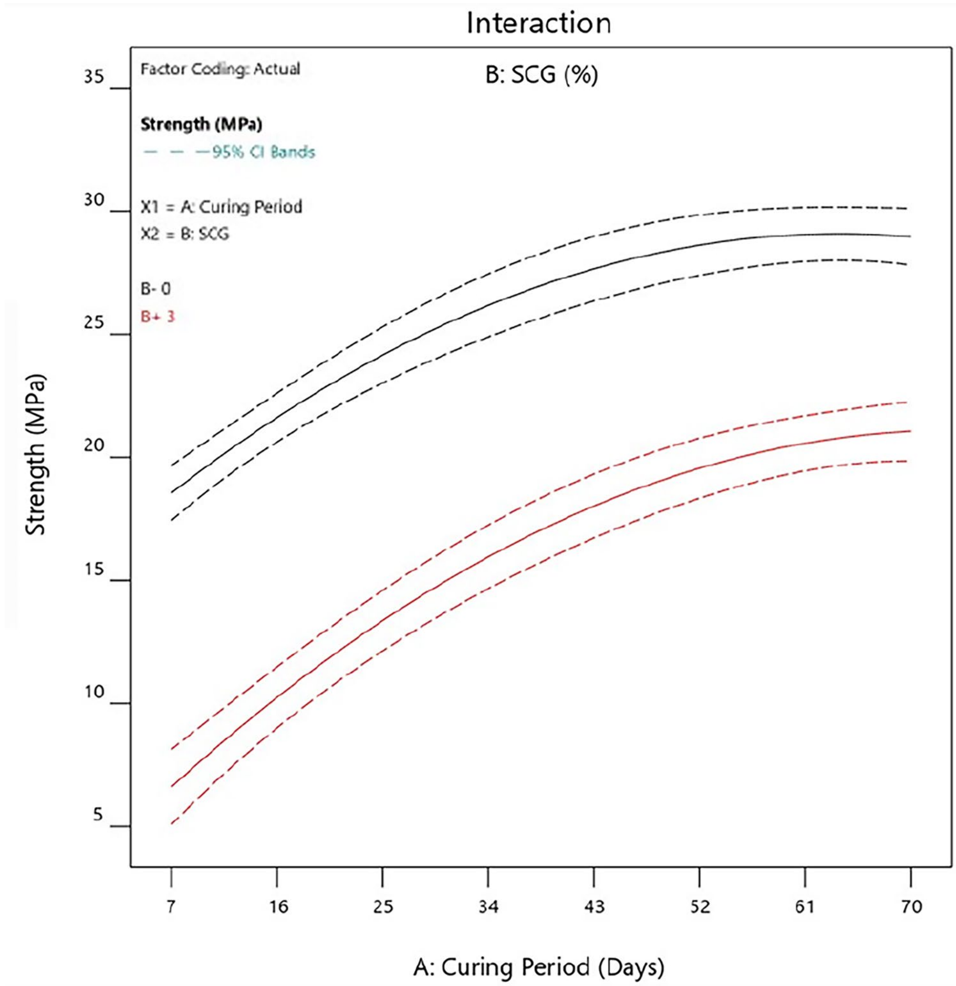

Fig. 9 Interaction plot of SCG content and curing days as a response to compressive strength

in strength until it reaches 3%. In terms of the curing period, a steady increase in compressive strength can be observed with the increase in curing periods. It is observed that the compressive strength experienced the highest increase from 7 to 28 days of curing time. Upon reaching the 28-day curing period, the rate of strength gain in the cement mortar decreases, resulting in a lower strength after 52 days of curing. These findings are similar to previous literature where concrete is known to achieve 65% of its strength during 7 days of curing and 99% strength during 28 days of curing [64]. Therefore, even if there is strength gain after 28 days of curing, the rate of strength gain would decrease significantly.

The interaction plot shows that both the curing period and SCG content did not cross, hence no interaction between these two parameters. The perturbation plot identifies the most influential parameter on the compressive strength of the SCG cement mortar. From the perturbation plot, it can be seen that SCG content is steeper than that of the curing period, which means that the compressive strength is greatly affected by SCG content in comparison to the curing period. Figure 11 shows the one-factor plot of SCG content and curing period versus compressive strength. It can be observed that SCG content peaked at around 1%, while for the curing time apparently the plateau is not reached until around 65 days. This value is reflected in the value of SCG content and curing period that correspond to optimum compressive strength. According to Lee et al. [45] mortar containing a higher SCG mix (e.g., 6%) has higher water absorption (> 5%). Thus, it is not recommended to add a higher content of SCG in cement mix, since no gain in strength was observed at higher SCG content (> 2%). Moreover, cement mortar with higher water content which is associated with higher SCG content (e.g., 6%), may suffer from shrinkage cracking issues.

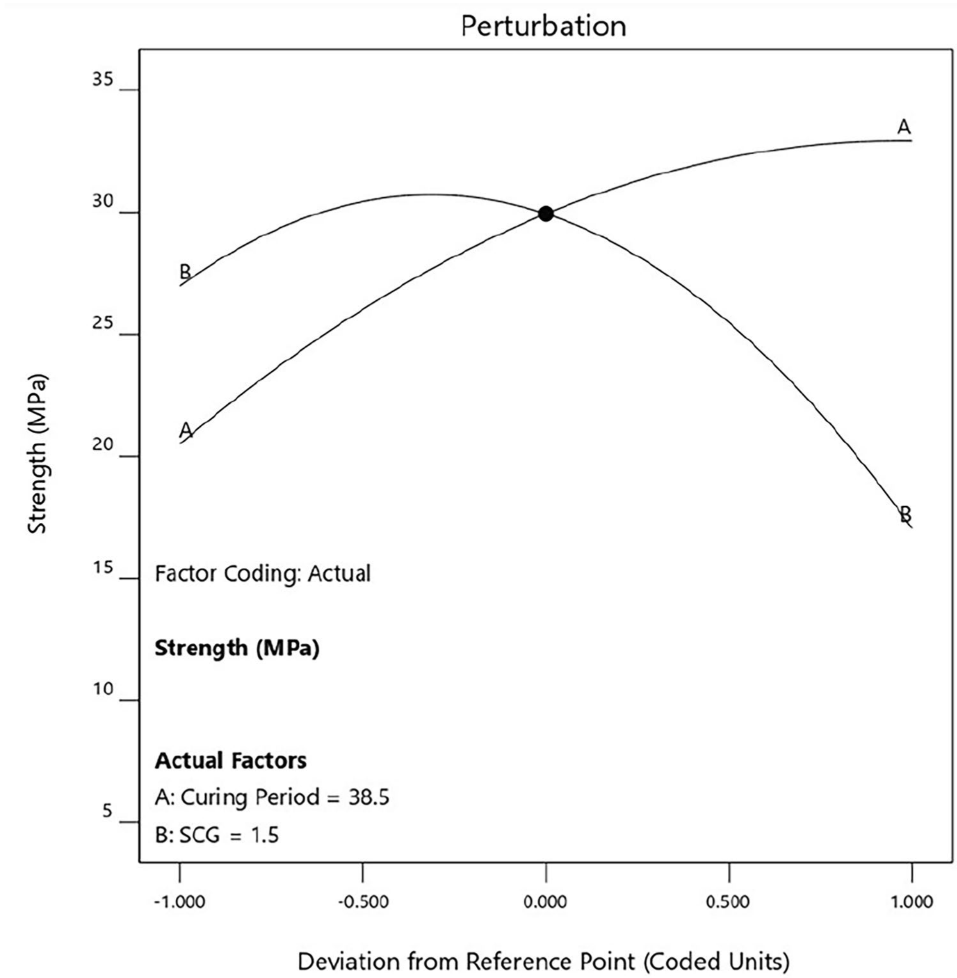

Fig. 10 Perturbation plot of the SCG cement mortar. **A** Curing days; **B** SCG (%)

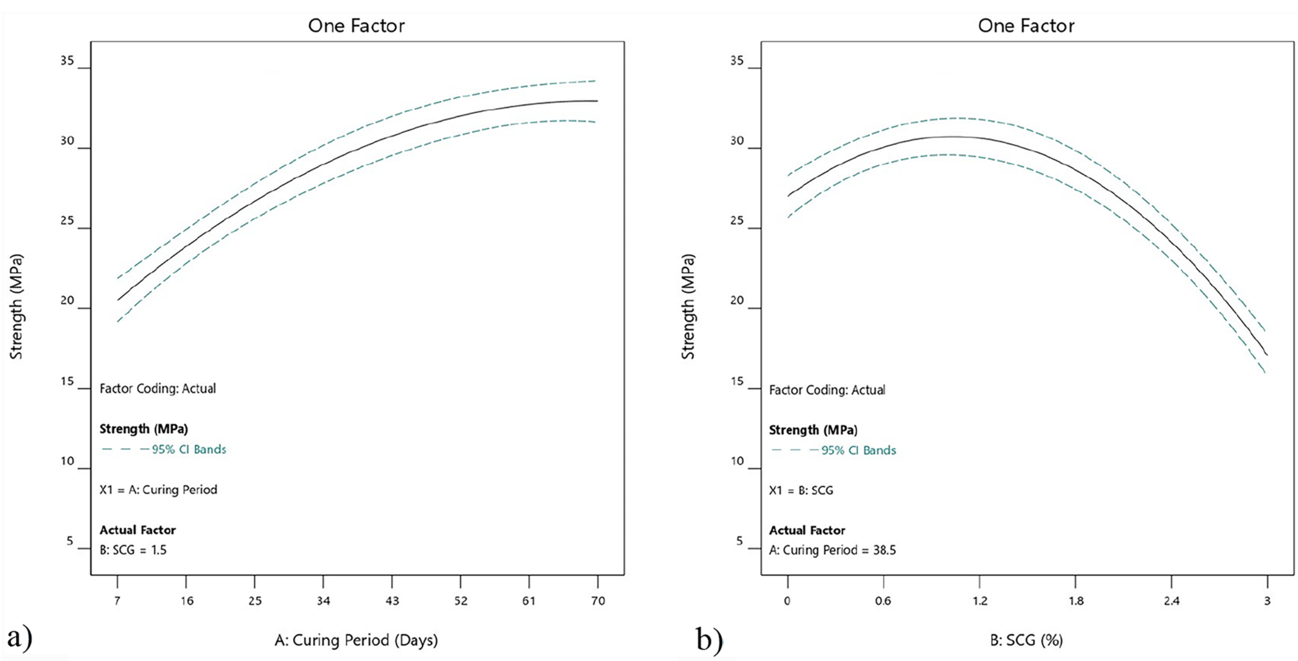

Fig. 11 One factor plot of the model. **A** Curing period versus compressive strength. **B** SCG content versus compressive strength

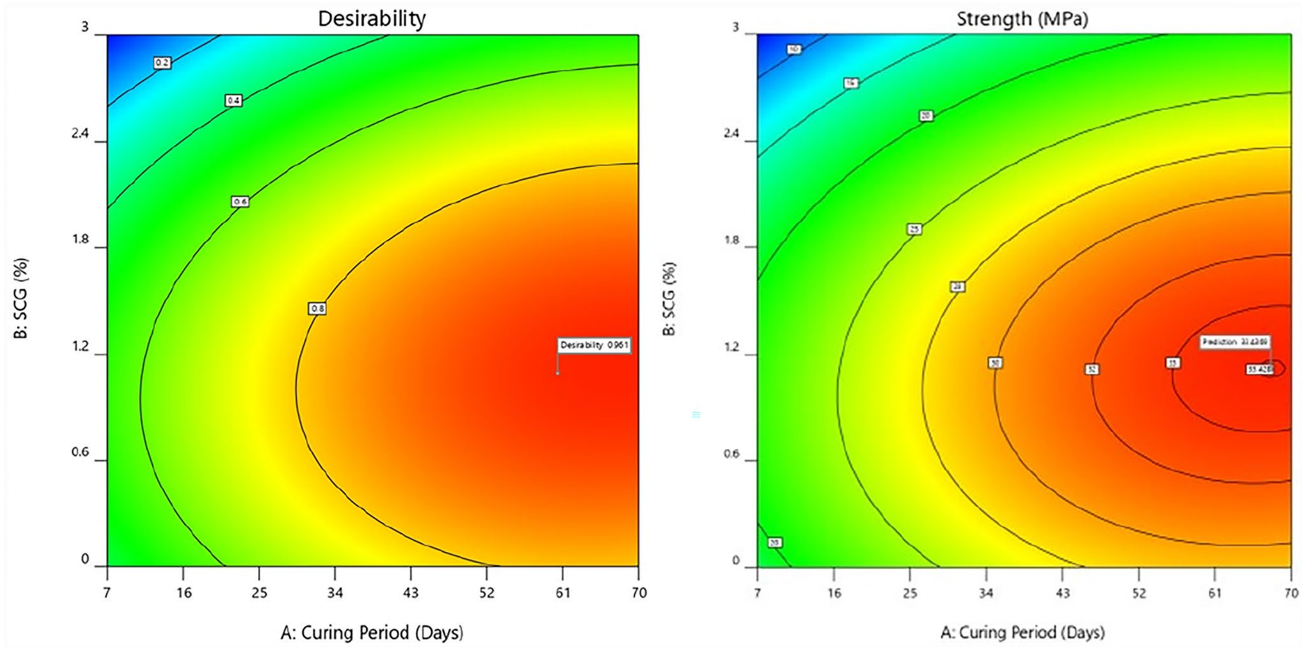

Fig. 12 Optimization of 2D contour plot to determine superlative parameters for ultimate strength

3.4 Optimization study of SCG cement mortar

The optimization of the model was performed by setting the curing period in the range of 7 days to 70 days, while the SCG content was set in the range of 0% to 3%, to maximize the compressive strength. From the 2D contour plot (Fig. 12), the highest compressive strength is determined to be at 33.44 MPa. This optimum point was achieved using 1.12% SCG in the cement mortar mix with a curing period of 68 days. The desirability of this optimum point of 0.97 is close to unity representing the closeness of this prediction to its ideal value.

A validation run was performed for the optimum point using three different samples containing 1.12% SCG and 68 days of water curing. The average compressive strength value obtained from the three samples is around 32.40 MPa. The difference between the validation value and the model optimization value is around 3.12%. The difference is considered acceptable and the variation between the validation results and the optimization results could be due to uncontrolled circumstances such as the presence of extremely hot and dry weather conditions. The model is considered valid and may be used to predict the compressive strength of cement mortar with SCG additive.

The compressive strength of control cement mortar without SCG after 70 days of water curing is determined to be at 28.77 MPa. The RSM optimization predicted that the cement mortar mix containing 1.12% SCG and undergoing 68 days of curing may achieve up to 33.44 MPa of compressive strength. Though validation results were slightly lower at 32.40 MPa, the optimized mix still proved superior over the cement mortar without SCG additive. Overall, comparing the controlled cement mortar mix without SCG additives, there is an increase of 12.62% observed in the RSM-optimized cement mortar containing SCG content. Hence, the optimization studies in this work have demonstrated that the highest strength of cement mortar can be obtained using response surface methodology.

4 Conclusion

The SCG additive in this study can increase the compressive strength of cement mortar from 28.77 MPa for the control sample without SCG to 32.40 MPa for the ones with SCG. The SCG surface morphology was found to be irregularly shaped and prone to moisture absorption. Chemical composition analysis shows that SCG is a material high in carbon content with at least 66.85%. The workability of the cement mortar decreased with the increased volume of SCG in the mix due to the moisture-absorbing nature of SCG particles. The response surface model was used to optimize the SCG content and curing time of SCG cement mortar. From the optimization model, the highest compressive strength of SCG cement mortar was found at 33.44 MPa using 1.12% SCG and a curing time of 68 days. Experimental validation for the optimum point yielded a compressive strength of 32.40 MPa, which has around 3.12% deviation from the model prediction. The control cement mortar with no SCG additives cured at 70 days possesses 28.77 MPa compressive strength, while the RSM-optimized mix containing SCG presented at least a 12.62% improvement in compressive strength after 68 days of curing. The findings in this work proved that SCG may potentially be used as part of the concrete constituents to increase its compressive strength. Application of SCG in the cement mortar as additives provides several benefits to the construction industry beyond just improving the strength of the cement mortar, that is the high accessibility to SCG wastes, the low reutilization costs, and environmentally friendliness.

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Data availability Data for the optimization study is provided in the manuscript.

Declarations

Competing interests The authors have no competing interests to declare that are relevant to the content of this article.

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