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Recycling Of Chromite Waste for Concrete: Full Factorial Design Approach

Mesci, B.[1*] and Elevli, S.[2]

[1] Department of Material Science and Engineering, Ondokuz Mayis University, 55139 – Kurupelit - Samsun, Turkey

[2] Department of Industrial Engineering, Ondokuz Mayis University, Samsun, Turkey

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ABSTRACT: Recently full factorial design approach has been used to assess the recycling potential of a given waste. The objective of this study is to identify the significant factors and interactions involved in maximizing compressive strength of concrete when chromium waste is used as admixture. In this respect, experimental factors at two levels, which are admixture type (chromite/ chromite+ superplastizer), admixture percentage (5-15%) and cure duration (7-28 days), are selected as possible candidates affecting the compressive strength. The modeling technique presented in this paper is based on 2^3 full factor experimental design and can easily be implemented to see the effect of any input factor on a given response variable. According to the full factorial analysis, at the 5% percentage level when Naphtalene sulfonate is added to concrete with chromite waste, the compressive strength will be good enough after 28 days. The results demonstrate that chromite waste after neutralization can be used in construction industry.

Key words: Chromite waste, Experimental design, ANOVA

INTRODUCTION

In today's world, regulations on waste elimination make waste management a significant problem for the public authorities. To reduce pollution and disposal of wastes, there is an urgent need to maximize recycling and reuse (Singh and Garg, 1999; Whitlock and Galitz, 1997). In this scope, many research results have been reported on incorporating various industrial wastes into construction materials (Aubert *et al.,* 2004; Papakonstantinou and Tobolski, 2006; Batayneh *et al.,* 2007; Gennaro *et al.,* 2008; Federico and Chidac, 2009). Since studies concerning the use of industrial waste materials in concrete production contribute to sustainable development, the investigations on alternative waste materials usage tend to continue in future (Huang *et al.,* 2009; Meddah and Bencheikh, 2009; Topçu *et al.,* 2009).

The use of chromium compounds in many industries because of its high corrosion resistance and hardness is a source of a large quantity of waste. Since acidity and significant concentration of chromium compounds in the wastewater make chromium industry of high environmental concern, technology of chromium wastewater neutralization has been commonly applied. This technology is based on detoxification of waste, which is done by reducing chromium(VI) to chromium(III) and then, using sodium or calcium hydroxide, insoluble chromium(III) hydroxide is precipitated (Bojanowska, 2002; Bielicka *et al.,* 2005). At the same time other metal hydroxides are co-precipitated. Dewatered sludge is then stored at landfills. However, storage should not be considered as final step from the environmental point of view. The recycling opportunities should still be investigated. Although the use of different types of wastes in cement-based materials has become a common practice in concrete industry, the potential of reusing the chromite waste in construction material is less studied. The aim of this study is to investigate the recycling potential of chromium waste after neutralization in the area of construction industry to reduce the demand on land for disposing. Since one of the most important measurements of the performance and quality of cement is compressive strength, the goal has been chosen to maximize it by obtaining appropriate factor- level combinations. Full factorial design approach has been utilized for this process.The paper is organized as follows: Section 2 describes experimental procedure, including full factorial design and preparation of materials used in this study. Section 3 describes data analysis and related results. Finally, Section 4 gives the concluding remarks.

MATERIALS & METHODS

The materials consist of Ordinary Portland cement, chromite waste after neutralization, and super

*Corresponding author E-mail: basakm@omu.edu.tr

plasticizer. The cement used in this study is commercial grade ASTM Type I ordinary Portland cement (OPC), which is produced as CEM I cement in Turkey. This type of cement is most widely used for construction industry in Turkey. The chemical composition and physical properties of the cement are given in Table 1.

Table 1. Physical properties and chemical composition of the cement

Properties	**Cement**
Initial setting time (min)	164
Final setting time (min)	225
Blaine specific area (cm^2/g)	3235
Specific gravity	3.08
Fineness	14.6
>32 lm, %	0.10
>90 lm, %	wt %
Chemical compositions (%)	
SiO_2	18.61
Fe_2O_3	3.23
Al_2O_3	5.28
CaO	62.83
SO_3	2.74
K_2O	0.85
MgO	1.02
Na_2O	0.48
P_2O_5	0.07
MnO	0.09
TiO_2	0.24
LOI*	4.14
Others	0.42
Main compounds	
C_3S	67.34
C_2S	6.70
C_3A	8.56
C_4AF	9.75

The chromite industry waste sample is obtained from the Kromsan Plant of Mersin, Turkey. This plant produces sodium chromate by processing chromite ore ($FeCr_2O_4$). The chromite industry waste sample is taken from the plant after the neutralization process. After the second filtration process of the solid waste in the production of sodium monokromat, the amount of CrO_3 is measured as 0.1%. The chemical composition before neutralization for chromite industry waste is given in Table 2. The total amount of SiO_2, Al_2O_3 and Fe_2O_3 was 61.75% which was lower than the minimum requirement (70%) specified by ASTM C 618 for pozzolanic material (ASTM C 618, 2001).

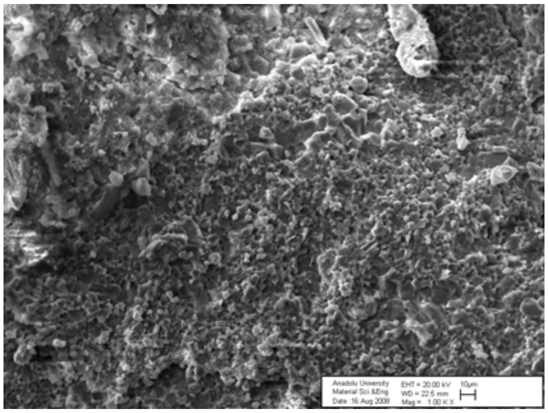
A natural river sand and crushed limestone were used as fine and coarse aggregates. The properties of aggregates are in conformity with the ASTM C 33. A super plasticizer (SP) of sulfonated naphthalene formaldehyde type was used meeting standard specifications ASTM C 494 Type F. The water to cement ratio (W/C) was kept constant at 0.58. The water used was distilled.

Table 2. Chemical composition (wt %) of the chromite industry waste samples

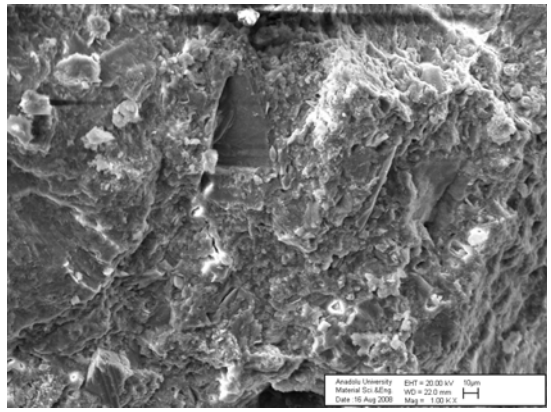
Chemical compositions (%)	**Chromite Waste**
SiO_2	1.08
Fe_2O_3	43.40
Al_2O_3	20.90
CaO	0.18
MgO	14.30
Cr_2O_3	12.68
LOI*	4.47
Others	2.99

Super plasticizer and naphthalene sulfonate were added to the mixing water, which was then mixed with the cement. Mixing was carried out at the room temperature. Cube specimens (100x100x100 mm^3) were cast in steel moulds. Conventional destructive test methods were used to determine the strength of concrete (ASTM C 403). The minimum values of the required compressive strength of the OPC are obtained at 7 and 28 days under the specific mixture and testing conditions of 264.4 kg/cm^2 and 312.5 kg/cm^2, respectively. Furthermore, the compressive strength of the OPC with superplasticizer is obtained at 7 and 28 days under the specific mixture and testing conditions of 347.5 kg/cm^2 and 382.3 kg/cm^2, respectively. Fig. 1 demonstrates SEM micrograph of samples, which consist of cement + super plasticizer (a), cement + chromite industry waste (5%) + super plasticizer (b) , and cement + chromite industry waste (15%) + super plasticizer (c) for a period of 28 days.

Variations or additions to the conventional concrete formulation are known as admixtures. Admixtures often exhibit enhanced performance properties and have been used fairly commonplace for many years (Correia *et al.*, 2009; Galetakis and Raka, 2004). The selection of appropriate admixtures is very important criterion to achieve the desired specifications for concrete. Since the real life applications are mostly based on trial-error methods, the optimization using mixture designs and experiments gives many advantages to reach targets. However, in the traditional approach of experimentation for investigating the different features of concrete such as compressive strength, at a time, one factor is kept varying, and all the other factors are kept constant. Thus, it is a laborious, time and material consuming approach.Beside these drawbacks, the calculated optimum conditions may not be a true optimum if the interactions between the factors are present. To study the experimental factors and its interactions simultaneously, factorial experiments have been



(1-a)



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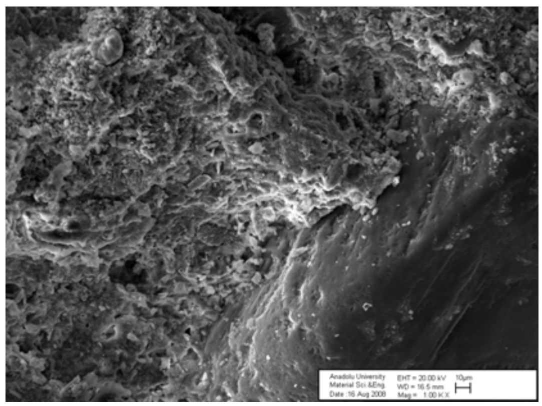

(1-c)

Fig. 1. SEM micrograph of samples with cement + super plasticizer (a), cement + chromite industry waste (5%) + super plasticizer (b), and cement + chromite industry waste (15%) + super plasticizer (c) for 28 days

recently used (Hýnýslýoðlu and Bayrak, 2004; Saikaew, 2009; Saikaew, 2007). Full factorial experimental design investigates the effects of two or more factors simultaneously at the levels tested on the output response such as compressive strength. There are also recent studies regarding with this topic (Sonebi *et al.*, 2000; Türkmen *et al.*, 2008; Yahia and Khayat, 2001; Mandal and Roy, 2006). A 2^3 factorial experiment is applied to estimate the effects of three factors on the compressive strength. The chosen factors at two levels are *A*: Admixture Type, *B*: Admixture Percentage, and *C*: Cure Duration as seen in Table 3.

Table 3. Experimental factors and levels

Factor	Low Level (-1)	High Level (+1)
(A) Admixture Type	Chromite waste after neutralization	Chromite waste after neutralization + Naphtalene sulfonate
(B) Admixture Percentage (%)	5	15
(C) Cure duration (days)	7	28

RESULTS & DISCUSSION

The two-level statistical design for three independent variables consists of eight factorial points (2^k=8) where each variable fixed at two different levels. The compressive strength (as a response variable) is assumed to be linearly dependent on the level of each independent factor. The experiments are repeated 3 times to increase the reliability. Table 4 shows the experimental design and related response variable.The effect of each experimental factor can be defined as the change in response when the factor changes from one level to another level such as from high to low level. An interaction is the variation among the differences between means for different levels of one factor over different levels of the other factor. For each effect and interaction, the null hypothesis that the effects are equal to zero was tested by using the Student's *t* statistic. Table 5 shows the estimated effects and coefficients. The *t*-tests reveal that all the main effects and the interactions except *AC* are significant at 5% level.

A normal probability plot of the standardized effects to determine the statistical significance of both main and interaction effect is seen in Fig. 2. The effects, that are not significant, will fall along a line; on the other hand, the significant effects will stray farther from the line. Thus, it is clearly understood that all the main effects and interactions except *AC* are significant statistically. Since *B* and *BC* lie on left hand side of the line, their contribution has a negative effect on the model. The reverse is true for the rest of the significant effects lie on right hand side. The factor Admixture Type (*A*) appears to have a largest effect because it lies furthest from the line. In addition to t- test, analysis of variance (ANOVA) is carried out to test the null hypothesis that main effects and the interactions (2-way and 3-way) are equal to zero, respectively. The small *P*-values (<0.05) mean that not all the main effects and interactions are zero at the 5% significance level. In other words, there is reasonably strong evidence that at least some of the main effects and interactions are not equal to zero. Table 6 gives the results of ANOVA test.

A mathematical model describing the main influence and interactions of various factors on a given response variable can be defined by using full factorial analysis. The linear model associated with a two-level statistical design in case of three independent variables (*A*, *B* and *C*) is expressed as in Eq. (1):

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC + \beta_{123} ABC \quad (1)$$

Third column of Table 5 gives the estimated coefficients of the linear regression model. The *AC* interaction, which seems to be insignificant based on the student's t-test, is eliminated from the model expressed in Eq (1). The reduced model, which includes the effects determined as significant, is defined in Eq. (2).

$$Y = 286.12 + 36.22 \cdot A - 14.87 \cdot B + 22.88 \cdot C + 4.37 \cdot AB - 13.27 \cdot BC + 3.47 \cdot ABC \quad (2)$$

A negative sign of a given parameter indicates that the response decreases with the increase in the value of the parameter. This means that (+1) level of factor *B* (admixture percentage) decreases the compressive strength. The magnitude and sign of *A* indicates that the contribution of this factor to compressive strength is 36.22 kg/cm^2 for the high level (+1). Similar explanations can be done for the other terms. The contribution of the interaction terms to the model seems rather low according to their coefficients. Since the sign of main effects (*A*, *B* and *C*) are calculated as (+), (-) and (+) respectively, the sign of *AB* and *ABC* interactions should be (-). This inconsistency can be passed over by eliminating these terms from the model. Thus, the new derived (simplified) model can be written as in Eq. (3).

$$Y = 286.12 + 36.22 \cdot A - 14.87 \cdot B + 22.88 \cdot C - 13.27 \cdot BC \quad (3)$$

According to interaction *BC*, the effect of factor *C* on response depends on the levels of factor *B*. That

Table 4. Matrix design and experimental data

Run No	A	B	C	Compressive Strenght (kg/cm^2) I	II	III
1	-1	-1	-1	233.5	236.7	239.6
2	+1	-1	-1	301	308	310
3	-1	+1	-1	216	218	221
4	+1	+1	-1	295	289	291
5	-1	-1	+1	293	305	307
6	+1	-1	+1	365	353	360
7	-1	+1	+1	235	257	237
8	+1	+1	+1	329	335	332

Table 5. Estimated effects and coefficients

Term	Effect	Coefficient	SE Coefficient	T	P
Constant		286.12	1.246	229.67	0.000
A	72.43	36.22	1.246	29.07	0.000
B	-29.73	-14.87	1.246	-11.93	0.000
C	45.77	22.88	1.246	18.37	0.000
A*B	8.73	4.37	1.246	3.51	0.003
A*C	0.90	0.45	1.246	0.36	0.723
B*C	-13.27	-6.63	1.246	-5.32	0.000
A*B*C	6.93	3.47	1.246	2.78	0.013
S = 6,10304 R-Sq = 98,85% R-Sq(adj) = 98,34%					

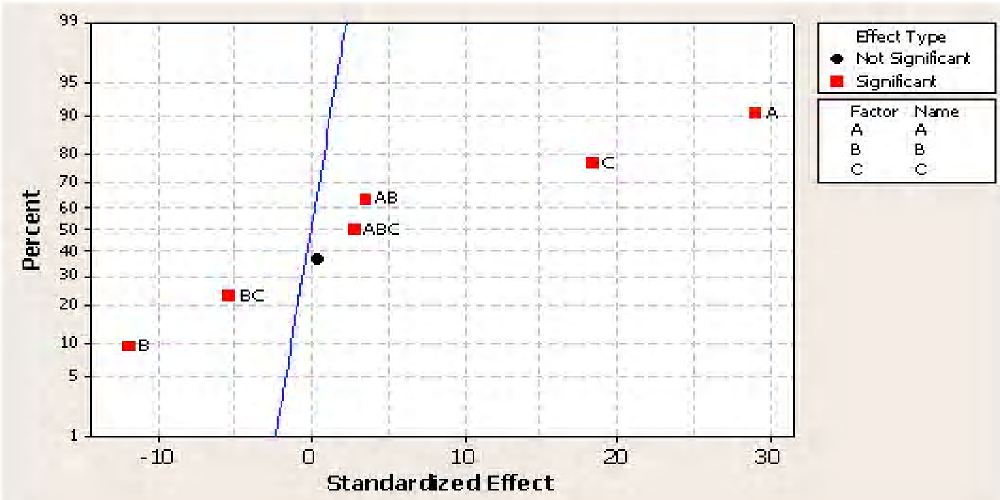

Fig. 2. Normal Probability Plot of Standardized Effects

Table 6. Analysis of Variance

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Main Effects	3	49351.5	49351.5	16450.5	441.66	0.000
2-Way Interactions	3	1518.5	1518.5	506.2	13.59	0.000
3-Way Interactions	1	288.4	288.4	288.4	7.74	0.013
Residual Error	16	596.0	596.0	37.2		
Pure Error	16	596.0	596.0	37.2		
Total	23	51754.4				

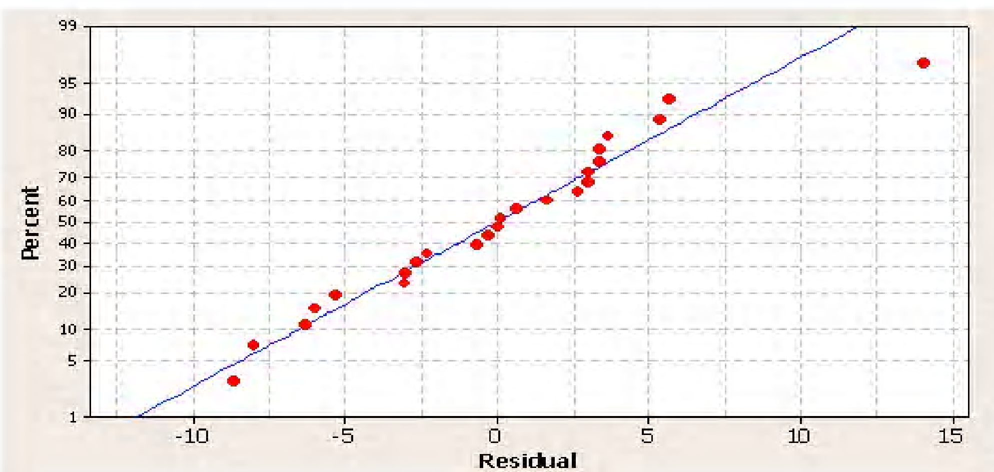

Fig. 3. Normal probability plot of residual values for compressive strength

is, compressive strength after 28 day for 5% admixture is higher than for 15%. The importance of the selected experimental factors varied according to the sequence: admixture type (Chromite waste after neutralization + Naphtalene sulfonate) > cure duration (28 days) > admixture percentage (5%). Based on this result, the compressive strength will be 373.36 kg/cm^2.

The analysis of a 2^k design assumes that the residuals are normally and independently distributed. A residual is the difference between an observation and its predicted value from the statistical model being studied. The normality assumption was checked by constructing a normal probability plot of the residuals as seen in Fig. 3. It can be concluded from the Fig. 3 that the experimental points are reasonably aligned on the suggested normal distribution.

CONCLUSION

As a result of increasing population and urbanization, great amounts of waste are produced. The disposal of waste such as chromite has become a severe social and environmental problem in Turkey as well the other countries. The possibility of recycling of waste from the construction industry is thus of increasing importance since it can help to reduce the demand on land for disposing. In this proposed study, the most appropriate factor - level combinations to obtain the highest compressive strength in case of chromite waste usage in concrete production as an admixture is investigated. In this respect, experimental factors at two levels, which are admixture type (chromite/ chromite+superplastizer), admixture percentage (5-15%) and cure duration (7-28 days), are selected as possible candidates affecting the compressive strength. According to the full factorial analysis, at the 5% percentage level when Naphtalene sulfonate is added to concrete with chromite waste, the compressive strength will be good enough after 28 days. The results demonstrate that chromite waste after neutralization can be used in construction industry.

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