

An experimental study on the recovery of the hardened cement from crushed end of life concrete

SOMAYEH LOTFI* AND PETER REM

Section of Materials and Environment, Faculty of Civil Engineering and Geosciences, Delft University of Technology
Delft, The Netherlands

* S.Lotfi@tudelft.nl

Abstract

In the C2CA concrete recycling process, autogenous milling of the crushed End of Life (EOL) concrete is a mechanical method to remove cement paste from the surface of aggregates. During autogenous milling, the combination of shearing and compression forces, promotes selective attrition and delivers a better liberation. In order to investigate the effects of shear and compression on the cement recovery and specify the importance of them, a new set-up is designed and constructed. This set-up permits aforesaid forces to be determined and controlled. For experimental design, the MINITAB 16 software was used and 13 different experimental runs based on varying shear and compression forces were conducted. After each experiment, the amount of cement recovery using XRF analysis, water absorption of the recycled aggregates and energy consumption during the process were measured. Results show that both shear and compression forces have influence on improving the cement recovery. With simple changes in the setting of an autogenous mill like bed height or residence time the need for high-cost secondary crushing during concrete recycling could be eliminated.

Keywords: concrete recycling, cement recovery, recycled aggregate

I. INTRODUCTION

Recycling End of Life (EOL) concrete is challenging for the building sector because of the competing constraints of low recycling process cost and high product quality. The C2CA process aims at a cost-effective system approach for recycling high-volume EOL concrete streams into prime-grade aggregates and cement. One of the main technologies considered within C2CA is autogenous milling. After crushing the EOL concrete, liberation of the cement paste is promoted by several minutes of grinding in an autogenous mill while producing as little as possible fine silica (Lotfi et al., 2014) and (Lotfi et al., 2013).

The conventional EOL concrete recycling circuit is composed of simple size reduction and classification for RA production. However, it does not deliver a high amount of liberated aggregate and cement paste (Kim et al., 2012). According to some studies (Florea et al., 2013) the liberation of the cement paste in the fine fraction will enhance by increasing the number of crushing processes. A secondary crusher can apply pure compression and bring higher cement recovery. However, using pure compression is not always economic and beneficial. It requires a substantial amount of energy and there is a high possibility of breaking aggregate into the

fine fraction.

The idea of using autogenous milling instead of the secondary crushing has a root in a fundamental principle of mineral processing. In mineral processing, one of the aims of comminution is liberation of valuable components separated from gangue. In an autogenous mill, internally created shear and compression forces produce a gentle attrition among particles. Therefore, surface layers, edges or corners from crushed EOL concrete can be removed (King, 2012).

In the present study, the influence of shear and compression forces on the cement recovery from EOL concrete was determined. Evidence is presented that the amount of cement recovery in the crushed fine fraction EOL concrete is influenced by both shear and compression forces. The aim of this investigation is to enrich our understanding of the importance of noted forces with respect to the cement recovery and also enable advances in the field of concrete recycling.

II. MATERIALS AND METHODS

Parent concrete and primary crushing

Table 1 shows the mix design of the parent concrete with the strength class of C30/37. After casting and six months curing of the parent concrete, a jaw

Table 1: Mix proportions of the concrete per m³

Component	Wet [kg]	Dry [kg]
CEM III/B	330	330
Sand 0,125-0,250 mm	74.72	74.72
Sand 0,250-0,500 mm	242.85	242.85
Sand 0,500-1 mm	242.85	242.85
Sand 1-2 mm	149.45	149.45
Sand 2-4 mm	93.4	93.4
Gravel 4-8 mm	373.61	373.61
Gravel 8-16 mm	691.18	691.18
Water	165	
Super plasticizer	0.27	
Total	2363	2198

crusher with the opening of 20 mm was used for the primary crushing. Figure 1 shows the particle size distribution of the crushed parent concrete.

Set-up for applying shear and compression force

A new experimental set-up with the purpose of applying controlled shear and compression on the bulk crushed concrete was designed and constructed. The schematic of the Shear - Compression Machine (SCM) can be seen in Figure 2. The SCM consists of a vertical cylinder for the application the compression force. A ring-shaped container is placed under the vertical cylinder which is connected to an arm. An electrical engine connected to the arm is applied to move the container back and forth. The effective surface area of the container is 0.12 m², and for each test it can be filled out with approximately 22 kg of crushed parent concrete.

Experimental design

In order to figure out the cumulative effects of two variables (shear and compression), it was decided to use Response Surface Methodology (RSM). Within RSM, Central Composite Design (CCD) is a popularly used method (Montgomery, 2008). As shown in Figure 3, a two-variable CCD is composed of 2²=4 factorial points, extended by 2×2 additional axial points and 5 centre points (t₀) (five replications). In general, for a k-variable CCD, the total number of simulated runs T is calculated by:

$$T = 2^k + 2k + t_0 \quad (1)$$

Repeating runs at the centre of the design introduces a check on variability and repeatability into the data, providing a means to eliminate noise in the experimental results. The general form of RSM with the second-order model is expressed as:

$$y = \beta_0 + \sum_i \beta_i x_i + \sum_i \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j + \epsilon \quad (2)$$

Table 2: Variables and their level for experimental design

Variable	Low level	High level
Compression: Force (KN)	1.2 KN	36 KN
Shear: Duration (min)	2 min	8 min

Where β_i represents the linear effect of x_i , β_{ii} represents the quadratic effect of x_i , β_{ij} represents the interaction between x_i and x_j and ϵ is the fitting error.

In this research, design and analysis of the central composite experiment were carried out using the MINITAB 16 software. The variable of compression was defined as the force exerted by the cylinder, whereas the variable of shear was represented by the duration of the back and forth motion of the arm. Both variables had two different levels (low and high, see Table 2). In line with Equation (1), the total number of simulated runs was 13 experiments.

Analysis of the experimental samples

After each experimental run, the particle size distribution of crushed concrete was analysed. The chemical composition of CEMIII/B used in the parent concrete was determined using XRF (model PANalytical-Epsilon 3x spectrometers) (see Figure 4). The XRF result of the cement shows the amount of SiO₂ and CaO, 27.86 mass% and 47.55 mass% respectively (See table 3). In order to calculate the cement recovery, XRF analysis for all 0-1mm and 0-0.5mm fractions was conducted. According to the primary weight of cement and silica (sand plus aggregate), and assuming that the main source of CaO in the parent concrete was cement (compare Table 3 and 4), the total mass of CaO in the parent concrete could be calculated. XRF analysis of the 0-1mm and 0-0.5mm fractions yields the mass% of CaO. Thus using the mass (gr) of the considered fractions, their total mass(gr) of CaO can be calculated. The cement recovery is then calculated based on the calculated mass of CaO in the considered fraction divided by the total mass of CaO in parent concrete.

The water absorption of the 4-16 mm fractions was determined to assess the amount of cement paste reduction and the energy consumption for each experimental run was calculated according to the torque measurement on the arm. Figure 5 shows the experimental set-up. A sensor was applied to determine the amount of torque and energy consumption during the experiments.

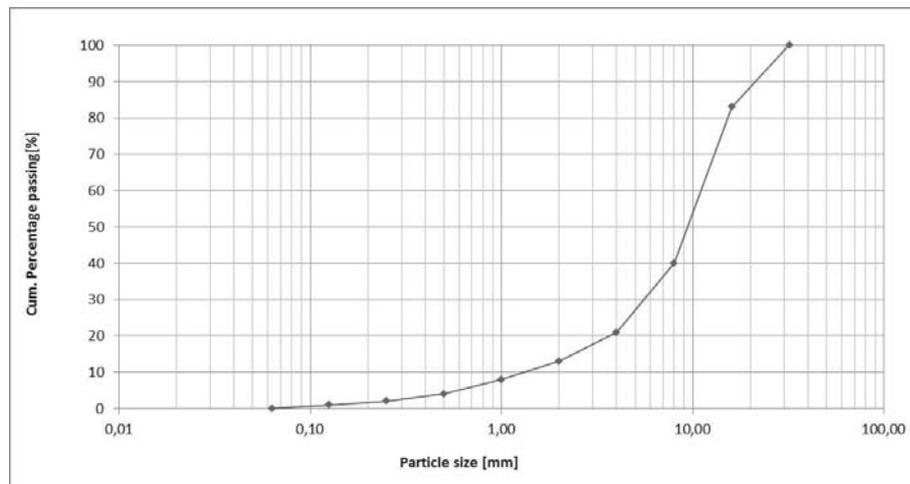


Figure 1: Particle size distribution of parent concrete after primary crushing

Table 3: XRF analysis of applied cement (CEMIII/B) in the parent concrete

Component	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	Cr ₂ O ₃
Mass%	7,85%	10,33%	27,86%	3,48%	0,47%	47,55%	0,72%	0,01%	0,00%
Component	MnO	Fe ₂ O ₃	CuO	ZnO	SrO	Y ₂ O ₃	ZrO ₂	SnO ₂	BaO
Mass%	0,17%	1,32%	0,01%	0,02%	0,10%	0,01%	0,03%	0,02%	0,04%

Table 4: XRF analysis of applied aggregate in the parent concrete

Component	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	TiO ₂	V ₂ O ₅	Cr ₂ O ₃
Mass%	0,13%	2,79%	94,83%	0,23%	1,30%	0,21%	0,13%	0,01%	0,01%
Component	MnO	Fe ₂ O ₃	CuO	ZnO	SrO	Y ₂ O ₃	ZrO ₂	SnO ₂	BaO
Mass%	0,01%	0,17%	0,01%	0,01%	0,00%	0,00%	0,02%	0,13%	0,00%

III. RESULTS AND DISCUSSION

Interpretation of the regression analysis

Experiments were performed according to the experimental plan and the results are given in Table 5 along with the results predicted by the model. Tables 6 and 7 show estimated regression coefficients for cement recovery in the 0-1 mm and 0-0.5 mm fractions, respectively. Using T-test and P-values regression analysis was carried out. In general, the larger the magnitude of T and the smaller the value of P, the more significant is the coefficient term (Montgomery, 2008). Considering Tables 6 and 7, the effects of the linear factors on cement recovery are highly significant ($P < 0.001$). A positive sign of a coefficient represents a synergistic effect while a negative sign shows antagonistic effect. In both tables, it can be seen that the linear terms of compression force and shear duration, the quadratic

term of force and the interaction term of force and duration have a positive effect on cement recovery. Those coefficients show that with an increase in the amount of force and duration the recovery percentage of cement will increase. Considering the regression coefficients, two regression equations for cement recovery in two different fractions 0-1mm and 0-0.5 mm result as following:

$$Y = 0.309341 + 0.079612X_1 + 0.039063X_2 + 0.014693X_1^2 - 0.008136X_2^2 + 0.014098X_1.X_2 \quad (3)$$

$$Y' = 0.201083 + 0.049825X'_1 + 0.030094X'_2 + 0.002352X'^2_1 - 0.005516X'^2_2 + 0.022357X'_1.X'_2 \quad (4)$$

Where Y is the Recovery of cement into the 0-1mm fraction and Y' is the recovery of cement into the 0- 0.5mm fraction. In both tables, the value of S

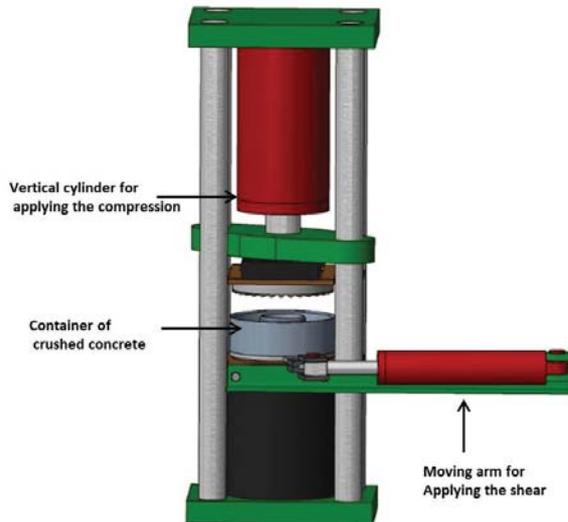


Figure 2: Schematic of Shear-Compression Machine(SCM)

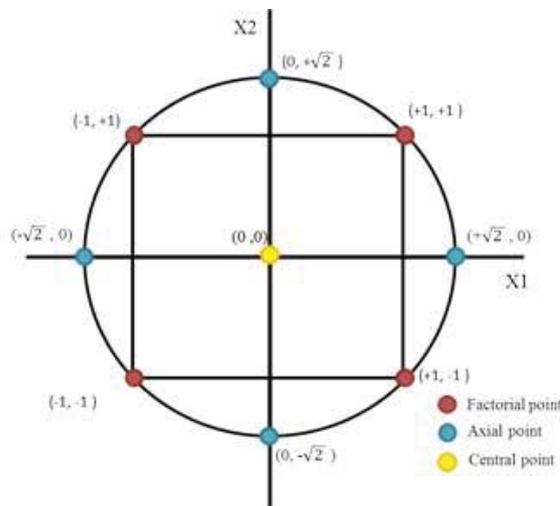


Figure 3: Layout of the experiments in a 2-factor Central Composite Design



Figure 4: XRF facility in the resources and recycling laboratory -TUDelft

(standard deviation) between the measured and predicted results shows that the equation adequately represents the relation between the response and significant variables. In particular, S is close to the experimental error of the data. The high value of ($R^2 = 97.06\%$, $R^2 = 93.12\%$) and (R^2 (adj) = 94.96% , R^2 (adj) = 88.20%) show high correlation between the observed and predicted values of response.

Main effect plot

A main effect is present when different levels of factor influence the response differently. It is created by plotting the response mean for each factor level. A line is drawn to connect the points for each factor and a reference line is also drawn at the overall mean (Greenfield and Metcalfe, 2008). When the line is not horizontal, there is a main effect present. Different level of the factor affects the response differently. The greater the difference in the vertical position of the plotted points, the greater is the magnitude of the main effect. The main effect of the parameters Force and timing on cement recovery from 0-1mm and 0-0.5 mm fraction are given in Figure 6A and 6B. Reference line in figures 6A and 6B is 0.3114 and 0.2001 respectively. From the figures, it is observed that both timing and force have a positive effect on the cement recovery. From the main effect plot, it is obvious that

force has slightly more influence.

Normal probability plot

The normality of the data can be checked by plotting the normal probability plot of the residuals. The normal probability plot is a graphical technique for assessing whether or not a data set is approximately normally distributed. Figures 7A and 7B show normal probability plot of residual values. Trends observed in those figures reveal well-behaved residuals. Based on this plot the residuals appear to be randomly scattered.

Interpretation of surface and contour plots

Contour and surface plots give a better understanding of the influence of variables and their interaction on the response. A contour plot provides a two-dimensional view, where all points having the same response are connected to produce contour lines. A surface plot provides a three-dimensional view that may provide a clearer picture of the response surface. Figure 8 shows the 3D or 2D plots relationship between two variables (force and timing) and properties like cement recovery, water absorption and energy consumption. According to the results, with increasing the amount of force and



Figure 5: Shear-Compression Machine

Table 5: Full factorial central composite design matrix of two factors, with experimental and predicted response (cement recovery)

Run Order	Force(KN)	Duration (min)	Recovery of cement in (0-1mm) Based on XRF results	predicted Recovery of cement in (0-1mm)	Residual	Recovery of cement in (0-0.5 mm) Based on XRF results	predicted Recovery of cement in (0-0.5mm)	Residual
1	6.30	2.88	25.00%	23.60%	1,40%	16.89%	15.40%	1,50%
2	18.60	5.00	30.93%	30.90%	0,00%	20.93%	20.10%	0,80%
3	18.60	8.00	35.03%	34.00%	1,00%	23.54%	22.60%	1,00%
4	1.20	5.00	23.31%	24.40%	-1,10%	14.27%	15.40%	-1,10%
5	36.00	5.00	41.66%	40.40%	1,30%	26.38%	25.30%	1,10%
6	18.60	5.00	30.57%	30.90%	-0,40%	19.53%	20.10%	-0,60%
7	6.30	7.12	27.82%	27.70%	0,10%	17.51%	17.40%	0,10%
8	30.90	2.88	33.13%	33.40%	-0,30%	20.19%	20.20%	0,00%
9	18.60	5.00	30.81%	30.90%	-0,10%	20.76%	20.10%	0,70%
10	18.60	2.00	25.38%	26.20%	-0,80%	15.54%	16.50%	-1,00%
11	30.90	7.12	38.77%	40.40%	-1,60%	25.28%	26.70%	-1,40%
12	18.60	5.00	30.90%	30.90%	0,00%	19.47%	20.10%	-0,60%
13	18.60	5.00	31.46%	30.90%	0,50%	19.85%	20.10%	-0,30%

Table 6: Estimated regression coefficient for cement recovery into the 0-1 mm fraction

Term	Coefficient	Standard error coefficient	T-Value	P-Value
Constant $= (X_0)$	0.309341	0.005281	58.577	0.000
Force(KN) $= (X_1)$	0.079612	0.005904	13.484	0.000
Timing(min) (X_2)	0.039063	0.005904	6.616	0.000
Force(KN)*Force(KN) $= (X_1^2)$	0.014693	0.008954	1.641	0.145
Timing(min)*Timing(min) $= (X_2^2)$	-0.008136	0.008954	-0.909	0.394
Force(KN)*Timing(min) $= (X_1 \cdot X_2)$	0.014098	0.011808	1.194	0.271

S(Standard error) = 0.0118084 R² = 97.06% R² (adj) = 94.96%

Table 7: Estimated regression coefficient for cement recovery into the 0-0.5 mm fraction

Term	Coefficient	Standard error coefficient	T-Value	P-Value
Constant $= (X'_0)$	0.201083	0.005462	36.816	0.000
Force(KN) $= (X'_1)$	0.049825	0.006107	8.159	0.000
Timing(min) $= (X'_2)$	0.030094	0.006107	4.928	0.002
Force(KN)*Force(KN) $= (X'^2_1)$	0.002352	0.009261	0.254	0.807
Timing(min)*Timing(min) $= (X'^2_2)$	-0.005516	0.009261	-0.596	0.570
Force(KN)*Timing(min) $= (X'_1 \cdot X'_2)$	0.022357	0.012213	1.831	0.110

S(Standard error) = 0.0122130 R² = 93.12% R² (adj) = 88.20%

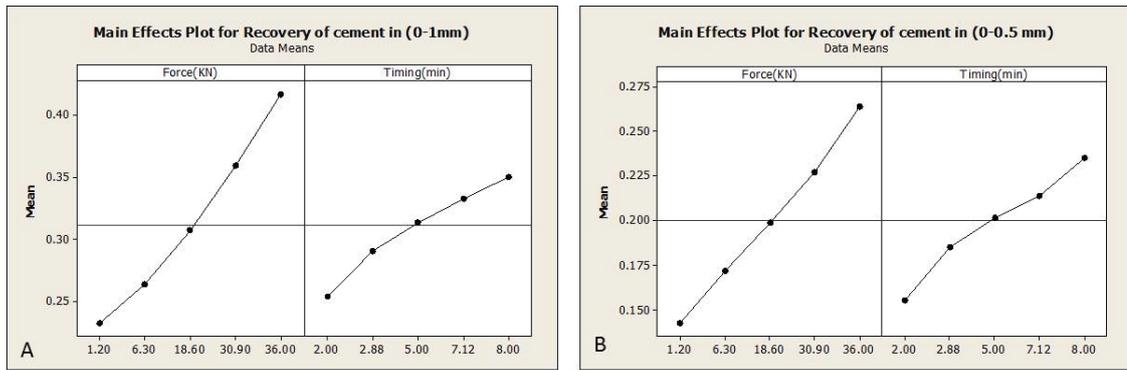


Figure 6: A: Main effects plot for recovery of cement in (0-1mm), B: Main effect plot for recovery of cement in (0-0.5mm). The duration of back and forth motion is indicated as timing

duration, the weight of 0-1 mm fraction and the cement recovery are increased. Recovery of cement is affected by both compression and shearing and it is increased to more than 40% in 0-1mm fraction (see Figures 8A and 8B). Decreasing the amount of water absorption in coarse fraction 4-16mm, by increasing the amount of force and timing, is another evidence to prove the reduction of cement paste on the surface of recycled aggregates (see Figures 8C and 8D). The energy consumption raises by increasing the amount of timing and force. However, according to Figures 8E and 8F even by using the highest amount of force and timing, the energy consumption is less than 700 kJ/ton (0.19 kWh/ton). It shows that the cost of autogenous milling could stay in a reasonable range during concrete recycling process.

IV. CONCLUSION

In the C2CA concrete recycling process, autogenous milling of crushed end of life concrete is used to increase the liberation of the cement paste. This research is carried out to understand how shear and compression, and the combined effect of them inside of an autogenous mill, influence the cement recovery. In order to simulate forces in an autoge-

nous mill in a controlled way, a new set-up was constructed. A central composite experimental design with the help of the MINITAB 16 software for predicting the results of 13 experimental runs was used. According to the regression analysis, the effect of shear and compression on the cement recovery for both 0-1 mm and 0-0.5 mm fractions was found to be strongly linear ($P < 0.001$). Comparing the main effect plots, force (compression) is slightly more effective than timing (shear). However, based on the achieved results, it is possible to replace the shear and compression with each other with the purpose of raising the cement recovery. Therefore, high amount of produced low-cost shear in an autogenous mill will eliminate the need for the expensive pure compression in a crusher. Variation in the strength of concrete could be compensated by simple changes in the mill feeding, the residence time and the bed height.

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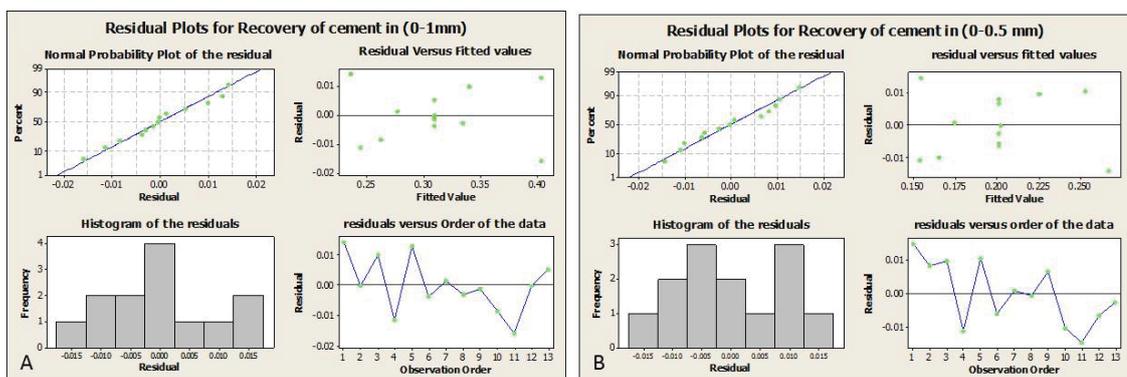


Figure 7: A: residual plots of cement recovery in (0-1 mm), B: residual plots of cement recovery in (0-0.5 mm)

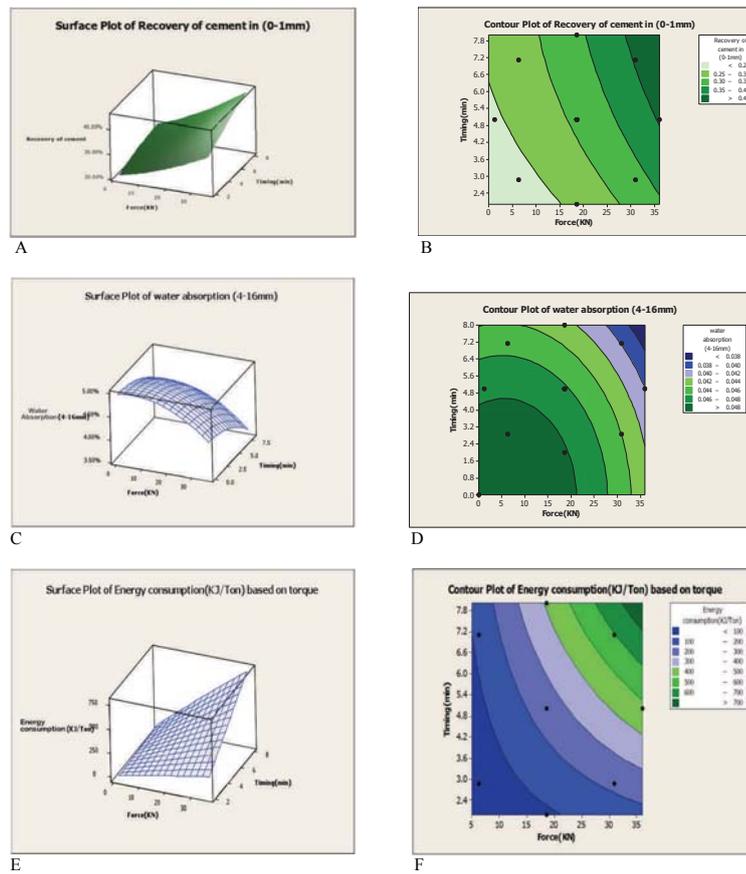


Figure 8: A and B: surface and contour plot for cement recovery in 0-1mm, D and E: surface and contour plot of water absorption 4-16mm, E and F: surface and contour plot for energy consumption during milling. In all figures duration of back and forth motion is indicated as timing.

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REFERENCES

Florea MVA, Brouwers HJH. Properties of various size fractions of crushed concrete related to process conditions and re-use. *Cement and Concrete Research*. 2013;52(0):11-21.

Greenfield, Tony, and Andrew Metcalfe. *Design and Analyse Your Experiment Using Minitab*. Hodder Arnold, 2006.

Kim KH, Cho HC, Ahn JW. Breakage of waste concrete for liberation using autogenous mill. *Minerals Engineering*. 2012;35(0):43-5.

King, R. Peter. *Modeling and simulation of mineral processing systems*. Elsevier, 2012.

Montgomery DCDaaoeJWS, 2008. *Design and analysis of experiments*: John Wiley & Sons, 2008.

Lotfi S., Dejab J., Rem J.P., Mróz R., van Roekel E. and van der Stelt H. (2014): "Mechanical recycling of EOL concrete into high-grade aggregates". *Resources, Conservation and Recycling*, vol. 87, 117-125.

Lotfi S, Deja J, Rem P, Mróz R, van Roekel E, van der Stelt H. A Mechanical Process for In Situ Recycling of EOL Concrete, SB 13, Singapore, 9 - 10 September 2013.