APC fly ashes stabilized with Portland cement for further development of road sub-base aggregates

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Abstract. Although waste-to-energy plants allow reducing the mass and volume of municipal solid waste (MSW) incinerated, an average around 30 % of the total content remains as bottom ash (BA) and air pollution control (APC) ashes at the end of combustion process. While weathered bottom ash (WBA) is considered a non-hazardous residue that can be revalorized as a secondary aggregate, APC fly ashes generated during the flue gas treatment are classified as hazardous waste and are handled in landfill disposal after stabilization, usually with Portland cement (OPC). However, taking into account the amount of APC residues produced and the disposing cost in landfill, their revalorization is an important issue that could be effectively addressed. As MSW can be incinerated producing bottom ashes (BA) or air pollutant control (APC) residues, the development of a mortar formulated with APC fly ash as secondary building material is a significant risk to the environment for their content of heavy metals. In this way, Design of Experiment (DoE) was used for the improvement of granular material (GM) formulation composed by APC and OPC for further uses as road sub-base aggregate. DoE analysis was successful in the modelling and optimization the formulation as function of the mechanical properties and APC amount. Consequently, an optimal mortar formulation (OMF) of around 50 wt.% APC and 50 wt.% OPC was considered. The OMF leachates and abrasion resistance have been analyzed. These results have demonstrated the viability of OMF as non-hazardous material feasible to be used as secondary aggregate. Moreover, it would be possible to consider the environmental assessment of a GM composed by ≈20 wt.% of OMF and ≈80 wt.% of WBA in order to improve mechanical properties and heavy metals stabilization.

1. Introduction

The urban waste increase is a direct consequence of the world's urban demographic growth. The solution for waste disposal relays in an incineration plant by means of revalorization processes, which not only reduce the waste volume produced but also acts as power generating system. The incineration process allows reducing between 70 and 90 % of the weight volume of the generated municipal solid waste (MSW). The waste production control involves a cleaning system to purify the resulting gases of the incineration, which varies depending on the nature of the waste. The correct treatment of urban waste is the incineration of all waste that cannot be recycled or directly reused, although this incineration process still requires a control in the two different by-products: ash (air pollution control, APC) and slag (bottom ash, BA) [1–5].

APC is obtained from the burning waste in one of the incineration stages and contains a high level of calcium resembling the pozzolanic behavior typically associated with cement. APC is used as complementary cement-like materials in the production of mortar for Portland base. A cement-like material, when used in conjunction with cement, contributes to the resistance properties of the material, whether it is through hydraulic or pozzolanic activity. On the other hand, BA is classified as a non-special waste, and it is an industrial waste principally of an inorganic. BA represents between 80 and 95 % of the total solid waste obtained in an incineration plant [6]. The slag produced from municipal solid waste incineration (MSWI) has a continuous particle size distribution in its heterogeneity, but a slight divergence can be observed in the bigger fractions. Once BA has been stabilized and homogenized, it can be valorized previous weathering stabilization during 2 - 3 months (weathered bottom ash, WBA), and it becomes a fine arid made mostly of concrete [7], ceramic materials, and glass [8] for construction purposes [9,10]. Because of the necessity of construction materials, the demographic growth has caused an increase in CO_2 emissions. Cement industry accounts around 8 to 15 % of total world emissions; hence, its optimization use is mandatory.

The aim of this investigation relays in the CO_2 emissions' reduction as a result of using the maximum amount of APC mixed with Portland cement to obtain the best mechanical, environmental, and economical properties for a mortar as a sub-base material for secondary roads application. Hence, different formulations have been studied in order to determine the optimum proportion. This new slag can be valorized, contributing to a decrease in the use of cement and in turn reducing CO_2 emissions.

2. Experimental procedure

2.1. Materials: Air Pollution Control (APC), Cement, and Centripor Retard 225

APC is considered a hazardous waste which must be properly stabilized before its final disposal in a landfill site. Although its composition may vary due to the technology used in the MSW incineration process, APC consists of fine particles, usually with a high content of heavy metals, organic compounds and chlorides [11]. Previous studies have shown that the amount of incorporated ash into cement is limited by the salt quantity, especially of chlorides [11–13]. A Portland cement (OPC) CEM I 52.5 R type has been used as well as an additive named Centripor Retard 225, from MC-Bauchemie which is a retardant of the setting time. The use of additive could leads to change some of the mortar properties, such as workability, malleability, and mechanical properties [11]. Accordingly, the workability (consistency) and the mechanical properties of the mortar formulations under study were evaluated.

2.2. Methods

2.2.1. Design of Experiments (DoE), X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD), mortar mixture.

A design of experiments (DoE) using the ''Design Expert 7.0'' software from Stat-Ease® was carried out. The DoE allows maximum information with minimum number of experiments. Furthermore, DoE lets to deduce which parameters have substantial influence in the optimal dosage and it also permits to quantify the effect on the parameters under study accordingly with the variation of the analyzed factors. The selected statistic design was *Historical data design* based on the Response Surface Methodology (RSM), which allows a further optimization of the mixture ratio. For all the studied responses, two factors were analyzed. The selection of these factors was made in accordance to a prior study as well as in the sake of obtaining the optimal dosage with the maximum amount of APC. These factors were the W/S ratio (mass of water per mass of solid, APC+OPC), and the APC percentage in the solid content (OPC+APC). This tool allows evaluating the influence of the factors on the mixtures in the range of study. For all the formulations, the total amount of the additive was fixed in 1 wt.% of the cement. Several responses were considered in order to obtain the optimal proportion. Table 1 summarizes the lowest and highest values for each factor under study. Thus, these values delimit the overall range of study for each property.

Table 1. Design summary of the studied factors.

Factor	Lowest level	Highest level
APC (%)	40.78	70.00
W/S	0.35	0.45

The analyzed properties for 22 formulations were consistency, and flexural (FS) and compressive strength (CS) at the age of 7 days. For better determine the effect of the variables for each response, a statistical design was developed. The analysis of DoE results is based on the analysis of variance (ANOVA). In this case, p-value has been used to interpret the obtained results. The p-value indicates whether the factor has a significant contribution to the model. A p-value lower than the level of significance ($\alpha = 0.05$) indicates significant contribution of the factor with a 95 % of confidence. Taking this into account, significant predictive models have been obtained for all the properties studied (see section 3.1). Subsequently, the optimization was conducted taking into account the criterion goals (CG) as follows (Table 2): incorporation of the maximum APC amount into the mortar and maintaining the highest mechanical properties.

APC were analyzed by means of X-ray fluorescence (XRF), using a Philips PW2400 X-ray sequential spectrophotometer to elucidate major and minor elements. X-ray diffraction (XRD) was performed in a Bragg–Brentano Siemens D-500 powder diffractometer with CuKα radiation in order to obtain information about the crystalline phases. Mortar mixtures were prepared accordingly to UNE-EN 1015-2 in a commonly mortar mixer. The cement was firstly mixed with water and the additive content to ensure the reactivity between them. Subsequently, APC was added for the properly mortar preparation. The resulting mortar was cast in polystyrene molds of 160 x 40 x 40 mm and homogenized by means of a vibration table. Every formulation was vibrated twice for 5 s with a pause of 30 s between them in order to remove the excess of entrapped air. Afterwards, the fresh molded samples were weighted before to be left in their molds for 72 h in a curing chamber at a constant temperature of 20 °C and a relative humidity of 95 % and the unmolded mortars were further allowed to cure in the same conditions up to 7 days. The samples were exhaustively supervised by control weighing during the curing period.

Table 2. Optimization criterions used in this study.

Constrains Name	CG	Limits		Importance
		Lower	Upper	
APC (%)	Maximize	40.00	70.00	5
W/S	Is in range	0.35	0.45	3
Consistency (mm)	Is in range	68.92	194.45	3
FS (MPa)	Is in range	1.168	5.276	3
CS (MPa)	Maximize	2.456	22.901	5

2.2.2. Characterization of the mortar: consistency, flexural and compressive strength, Los Ángeles test, and Leaching potential.

Concerning the experimental mixtures of the DoE, consistency was measured by the flow table test following the standard EN 1015-3:2007 and mechanical properties were evaluated at 7 days of curing time by following the standard EN 196-1. Flexural and compressive strength tests were the mechanical properties evaluated for each mixture.

In the case of the optimized mixture, in order to determine the abrasion resistance, Los Angeles test was carried out by using 10 kg of the optimal mixture and spinning between 500-1,000 times, using 11 abrasive load balls of 48.8 mm diameter and weight between 390 and 455 g.

In order to evaluate the leaching potential of heavy metals and metalloids in the optimal mortar, the dynamic EN-12457-4 leaching test [14]was carried out, which is used to categorize the solid wastes (inert, non-hazardous, and hazardous), according to the limits described in the criteria and procedures for acceptance of waste in landfills described in the European Landfill Directive. The EN 12457-4 leaching test procedure consisted of batch water leaching at L/S ratio of 10, in a 500 mL closed polyethylene reaction vessels, stirred at 3 rpm during a 24 h, at room temperature, with particles with size < 4mm. After 24 h of equilibration, the resulting suspensions were filtered through 0.45 µm membrane filters, and the obtained leachates were analyzed per duplicate using ICP-MS (Perkin Elmer Optima 3200 RL) and ICP-OES. The total digestion of samples for the analysis of the total content of heavy metals and metalloids (As, Ba, total Cr, Cu, Hg, Mo, Ni, Pb, and Zn) were carried out per duplicate.

3. Results and discussion

3.1. Design of Experiments (DoE)

The response surface methodology (RSM) succeed for finding an optimal dosage with a proper relation between APC (%) and W/S which improves the CG showed in the above mentioned Table 2. The models obtained for each response were statistically significant with lower *p-values* and small probabilities of being originated by noise. This was possible by taking into account the consistency, FS, and CS experimental results, and mixture proportions showed in Table 3. Therefore, the statistical models were developed by making multiple analyses with the help of the experimental values determined.

3.1.1. Consistency

A significant (*p-value*= 0.0005) statistical two factors interaction (2FI) model is obtained. The factors under study (APC % and W/S ratio) had a significant effect over the response. Figure 1 shows the consistency surface plot obtained. On one hand, an increase of APC percentage leads to a decrease of consistency. On the other hand, an increase of W/S ratio leads to an increase of consistency. Moreover, when both factors (APC and W/S ratio) are increased their combined effect is found to be lower than the expected from the sum of each one separately. Therefore, it should be concluded that there is a significant negative interaction between both factors (*p-value* = 0.0048) which explains this behavior. As it was expected, the amount of water in the mixtures increases the consistency value obtained and the increase of APC leads to a decrease of consistency value obtained due to their absorptivity capacity.

Table 3. Mixture proportions and experimental results.

Run	Factors		Consistency (mm)	FS (MPa)	CS (MPa)
	APC (%)	W/S			
1	40.78	0.35	103.75	4.279	22.901
2	52.24	0.41	183.08	3.444	10.595
3	50.00	0.45	194.45	3.116	9.046
4	70.00	0.35	101.45	1.242	3.001
5	40.78	0.35	131.21	4.450	21.612
6	70.00	0.41	121.75	1.168	2.483
7	70.00	0.35	88.09	1.333	2.456
8	60.00	0.38	147.55	2.382	5.998
9	70.00	0.45	134.42	1.256	2.492
10	55.00	0.35	90.96	2.492	7.023
11	70.00	0.45	138.58	1.592	3.145

12	40.80	0.35	106.66	5.183	19.798
13	52.20	0.38	110.18	2.918	8.661
14	50.00	0.38	108.06	3.732	11.198
15	70.00	0.42	110.60	1.330	2.943
16	40.80	0.35	96.24	5.276	18.129
17	70.00	0.42	107.95	1.409	3.225
18	70.00	0.42	98.16	1.466	2.841
19	60.00	0.4	116.80	1.250	3.884
20	70.00	0.42	68.92	1.955	6.298
21	55.00	0.39	125.11	3.015	6.780
22	70.00	0.42	98.16	1.466	2.841

The results derived from this analysis can be translated into a predictive mathematical model. The model can quantitatively anticipates the response within the operating range of controllable variables. In addition, it should be emphasized that it is possible to obtain some suitable formulations when a certain response is required. The model only shows the factors and interactions that are considered statistically significant. As it was aforementioned, in this case, a 2FI model describes the system. Hence, for the consistency results, the model can be written as it is shown in the following Equation (1):

$$\textit{Consistency}\ \ (mm) = -863.016 + 12.344\mathrm{APC} + 2829.520(\mathrm{W/S}) - 36.745(\mathrm{APC}\cdot(\mathrm{W/S})) \quad (1)$$

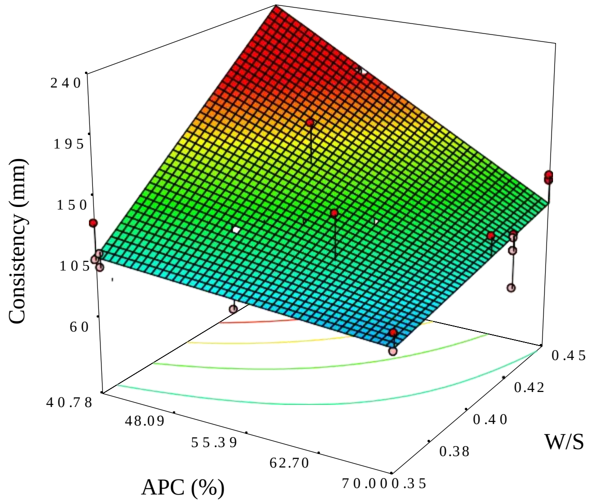

Figure 1. Surface plot of the consistency as a function of APC and W/S ratio.

3.1.2. Mechanical properties

Significant statistical models are obtained from *FS* and *CS* results at 7 days of curing, with the corresponding *p-values* of <0.0001 in both cases. Figure 2 depicts the surface plot for *FS* and Figure 3 shows the surface plot for *CS*. In addition, Equation. (2) and Equation (3) describe the mathematical model for each case, respectively. In the case of *FS* at 7 days, the best model is a reduced cubic model where there are cubic interactions between both factors (see Eq. (2)). In the case of *CS* at 7 days, the best fitted model is a reduced quadratic model. Besides, there are no interactions between the factors under study and the only significant effect is related with APC amount in the range under study (see Eq. (3)). In other words, the W/S ratio does not show significant effect in *CS* response in the range of study. This could be attributed to a higher effect of the selected range of the amount of APC in comparison with the selected W/S ratio for this study (see Table 1, levels of the study). In both cases, it is possible to confirm that an increase of APC in the mixture leads to a decrease of the mechanical properties at 7 days.

$$FS \quad (MPa) = -1099.150 + 22.767\text{APC} + 4894.293(\text{W/S}) - 115.206(\text{APC}\cdot(\text{W/S})) - 0.011\text{APC}^2 \\ - 3226.550(\text{W/S})^2 + 0.649(\text{APC}^2\cdot(\text{W/S})) + 45.984(\text{APC}\cdot(\text{W/S})^2) - 0.001\text{APC}^3 \tag{2}$$

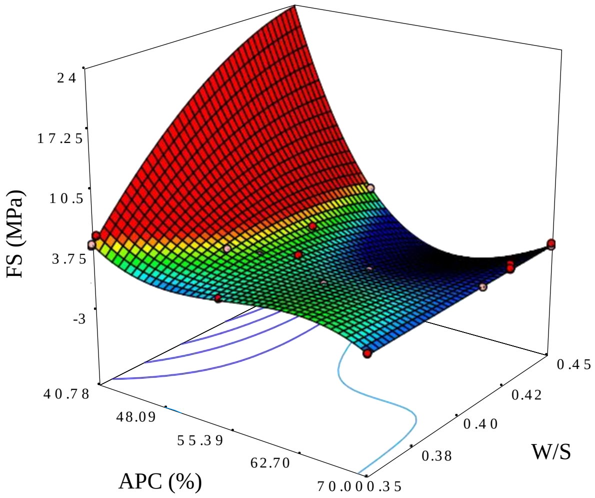

Figure 2. Surface plot of the *FS* at 7 days as a function of APC and W/S ratio.

$$CS \quad (MPa) = 111.248 - 3.173\text{APC} + 0.023\text{APC}^2 \tag{3}$$

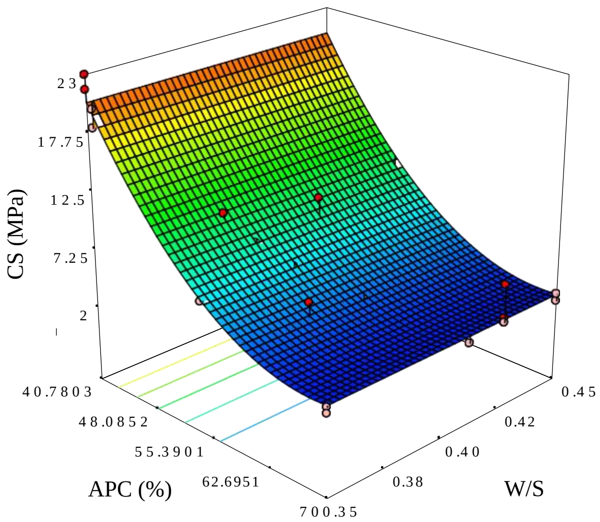

Figure 3. Surface plot of the *CS* at 7 days as a function of APC and W/S ratio.

3.1.3. Optimization and validation

Taking into account the statistical analysis presented above, a numerical optimization was performed in order to obtain the optimal composition of the mortar. As it was aforementioned, the basic idea of optimization entails a compromise between values by following the CG showed in Table 2. These selections were made because of the main purpose of this research was to obtain the proper mechanical properties of the mortars with a maximum amount of APC in the mixture. On this manner, it would be possible to retain the heavy metals after crushing the mortar for the development of a GM (80 wt.% of

WBA and 20 wt.% of the properly crushed mortar with the optimal dosage obtained in this section). The model was validated using the optimal mixture solution presented in Table 4.

Table 4. Optimal mixture solution based on CG.

Solution	APC (%)	W/S	Desirability
1	50.43	0.36	0.359

Thus, the optimal mixture solution was prepared and the responses were determined by following the procedure explained in previous sections. Consequently, Table 5 shows the obtained experimental results and also presents the predicted values and standard deviation for each response. Hence, it is possible to confirm that the experimental results were in agreement with those predicted by the model. Consequently, the validation demonstrates that the degree of fitting of the presented model is useful to further calculation of the responses.

Table 5. Validation of optimal solution based on CG.

Solution	Consistency (mm)		FS (MPa)		CS (MPa)	
	Experimental	Predicted	Experimental	Predicted	Experimental	Predicted
1	107.610	115.214 (103.370-127.060)	3.401	3.036 (2.527-3.545)	10.958	10.431 (9.477-11.385)

3.2. Characterization of the APC: X-Ray Fluorescence (XRF), and X-Ray Diffraction (XRD)

XRF results of APC are given in Table 6, where Ca^{2+} is the majority element, followed by Cl^-, Si^{4+} and S^{6+}. Moreover, XRD was performed, and the resulting components were NaCl, KCl, $CaCO_3$, CaO, CaCl(OH), $CaSO_4$, $Ca(OH)_2$, and MgO. The resulting salts are caused by the chloride derivatives from the combustion. Some studies [15–17] have shown a possible optimization with APC resides in pre-wash treatment to extract the maximum content of chlorides and sulfur [18].

Table 6. X-Ray Fluorescence APC results.

Compound	CaO	Cl	SiO_2	SO_3	K_2O	Na_2O	Al_2O_3	MgO	P_2O_5	TiO_2	Fe_2O_3	ZnO	PbO
Mean Wt (%)	48.35	8.85	6.64	6.24	4.33	4.28	4.02	1.73	1.36	0.86	0.79	0.72	0.12

3.3. Los Angeles test and leaching potential of the optimal mortar formulation

20 kg of the optimal formulation (Table 4) were conditioned for the following tests. The samples (prismatic specimens) were obtained by following the same procedure above-mentioned. The specimens were left in a curing chamber at a constant temperature of 20 ºC and humidity of 95 % up to 28 days. Consequently, the samples were crushed in order to obtain optimal mortar formulation as granular material (OMF-GM) in the proper manner for Los Angeles (LA) test and leaching test.

3.3.1. Los Angeles test

Generally, granulates with LA coefficient larger than 50 % are classified as bad quality granulates and cannot be used in construction sector. Also, those with an inferior value of 20 % constitute high performance granulates and are usually used as road sub-base [19]. A 10 kg of OMF-GM subsample for the particle size distribution values before and after LA test was used. The result of LA coefficient is 43-46 % for the optimal mortar formulation after 28 days of curing and crushing. On this manner, it can be considered that the OMF-GM is a medium-type granulate and it could be used for secondary roads.

3.3.2. Leaching potential

The leaching test results of OMF-GM and the threshold for non-hazardous materials stablished by the Catalan Government for Landfill disposal and the regulatory limit values for reutilization as secondary building material [20,21] are shown in Table 7. Most of the results are in agreement with non-hazardous limitation. Nevertheless, it is noticeable the chloride high content, therefore an APC prewashing would help to reduce the chloride content in the mortar and its pH.

Table 7. Results of the leaching test EN 12457-4 for OMF-GM and limit values. Results expressed in $mg \cdot kg^{-1}$.

Element	OMF-GM	Limit values			
		Landfill[a]			Utilization[b]
		Inert	Non-hazardous	Hazardous	
As	<0.10-0.20	0.5	2	25	1
Ba	22.80-67.76	20	100	300	-
Cd	<0.02	0.04	1	5	1
Cr_{total}	<0.01	0.5	10	70	5[c]
Cu	0.10-0.15	2	50	100	20
Hg	<0.001	0.01	0.2	2	0.2
Mo	0.06-2.12	0.5	10	30	-
Ni	<0.04-0.40	0.4	10	40	5
Pb	0.86-1.91	0.5	10	50	5
Sb	<0.04	0.06	0.7	5	-
Se	<1.00	0.1	0.5	7	-
Zn	0.54-9.50	4	50	200	20
Cl^-	76200	800	15000	25000	-
pH	12.4	-	≥6	-	-
K ($mS \cdot cm^{-1}$)	22.1	-	-	-	-

[a] Catalonian order number 2181/13.3.1996 [20].
[b] Catalonian order number 5370/30.4.2009, Decret 69/2009[21].
[c] Max. Cr(VI): $1 mg \cdot kg^{-1}$.

4. Conclusions

In the present research a statistical methodology was used with the aim to optimize a mortar formulated with OPC and APC fly ashes. The main goal of this research is the stabilization of APC fly by using the minimum quantity of OPC enhancing environmental criteria and diminishing the costs. The *Historical data design* based on *Response Surface Methodology* (RSM) was chosen to follow this research. The experimental responses used in the DoE were adequate for obtaining a fitted model accordingly with ANOVA results presented. The obtained model allows optimizing the dosage as a function of the required properties, i.e. mechanical properties, considering as better mechanical properties better stabilization of heavy metals. By following the aim of this research an optimal dosage was proposed and the model was validated. Furthermore, OMF was further studied as a potential granular material to be used as road sub-base aggregate. Leaching test and LA test of the OMF confirms its potential use as road sub-base aggregate. Moreover, it can be concluded that the use of the OMF-GM mixed with weathered bottom ash (WBA) in a 20 and 80 wt.%, respectively, would lead to a better GM as road sub-base aggregate. This assumption is based on the expertise of the authors working on this kind of materials for more than 10 years. The improvement is focused on two bullet points. On one hand, a decrease of the values in the leaching test is expected and attributed to the dilution of APC which presents the major heavy metals content in comparison with WBA. On the other hand, an improvement in LA test is expected because of the nature of WBA mainly compound by amorphous SiO_2 and $CaCO_3$, and also because of 33 % value of LA test presented elsewhere by the authors.

Therefore, this research seeds light into the optimization of granular material formulation which is suitable as road sub-base aggregate from mechanical and environmental point of view.

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