



Mix proportioning of concrete containing paper mill residuals using response surface methodology

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ABSTRACT

The use of paper-mill residuals in concrete formulations provides an alternative to landfill disposal. A response surface statistical methodology was carried out to model the influence on the slump and compressive strength of concrete containing paper-mill residuals with and without Class F fly ash replacement. The variables considered in this study included the water/cement ratio (w/c), paper-mill residual content and fly ash content to total cementitious material (FA/CM). The performance of the derived models to achieve good balance between the workability and compressive strength were further discussed by using the contour diagrams. The results showed that the compressive strength of concrete containing paper-mill residuals could be predicted from the referred slump value.

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1. Introduction

The pulp and paper industry generates several by-products in its manufacturing processes. These includes solid residue from wastewater treatment, ashes from the power boilers, and causticizing materials generated from chemical recovery associated with kraft pulping [1]. Among all, wastewater treatment-plant residuals constitute the largest solid waste stream generated by the pulp and paper industry. These wastewater treatment plant residuals consist predominantly of primary and/or secondary solids derived from primary and/or secondary treatment, respectively. Primary residuals are composed of wood fibers and an inorganic fraction (consisting of clay, calcium carbonate, titanium dioxide, and other materials used in pulp and paper production) collected during the separation of solids from the raw wastewater in primary treatment. Secondary residuals are mostly microbial mass collected by clarification following biological treatment [1–3]. The solids content of the water treatment plant (WTP) residuals usually ranges from 20% to 60%, depending in part upon the dewatering method applied [1].

In 1995, the pulp and paper industry in United State generated 5.8 million dry tons of wastewater treatment plant residuals, 2.8 million dry tons of which were managed in beneficial use applications, with the balance being disposed in landfills [3]. US Congress Office of Technology Assessment (USCOTA) reported that, of the

waste placed in on-site land-based units, such as landfills, surface impoundments, land application areas, and waste piles, the pulp and paper industry accounted for 35% of the total, and is the largest of any industry [4].

According to Naik's study in United State [5], saving on disposal cost of residuals by using of paper-mill residual in concrete is about \$0.375/cubic yard of concrete. If residuals were used for microfiber reinforcement of 20% of concrete produced in the US, there could be economic benefits of \$360 million for the concrete industry and \$30 million for the paper industry per year. Besides, several advantages on properties of concrete containing paper-mill residuals were found and summarized as follows:

- (i) By using proper amounts of fibrous residuals, water, and high-range water-reducing admixture (HRWRA), concrete mixtures containing the residuals were produced comparable to reference concrete mixtures (no residuals) in slump and compressive strength [5–7].
- (ii) With almost equivalent density, the splitting tensile and flexural strength of concrete containing residuals are higher than the reference concrete [5].
- (iii) The wood cellulose fibers in a residual significantly enhanced the freezing and thawing resistance of non-air-entrained concrete, bringing the resistance up to the level of air-entrained concrete [8].

Little studies have been carried out in the past to investigate the performance and establish the mix proportions of concrete containing paper-mill residuals, especially with the incorporation of

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fly ash as cementitious material. The fly ash is well-known for its advantages when incorporated in concrete. The use of fly ash confers economical benefit, improvements and reduction in temperature rise in fresh concrete, workability enhancement, and contribute to durability and strength in the hardened concrete [9,10]. Mohammed and Fang [11,12] reported that the use of Class F fly ash in producing concrete containing paper-mill residuals could result better resistance to both chloride-ion penetration and surface permeation than the Portland cement concrete.

Therefore, this used response surface methodology to model workability and compressive strength as well as, establishes the mixture proportion of concrete containing paper-mill residuals in combination with and without Class F fly ash as replacement. Use of response surface methodology in concrete technology has been highlighted in other publications [13–15]. The recommendations of this research will be beneficial for engineers and construction industries aimed to utilize paper-mill residuals based concrete.

2. Material and mixture proportions

2.1. Portland cement, fly ash, coarse and fine aggregates

Portland cement (PC) Type I, which conforms to the requirement of ASTM C 150-04 [16] with a specific gravity of 3.1 was used to produce the concrete in this research work. Class F fly ash (FA), conforming to the requirement of ASTM C 618-03 [17] with a specific gravity of 2.04 was used as partial replacement of PC. The fly ash was produced and received from Kapar Energy Ventures power plant in Malaysia. The chemical composition of PC and Class F fly ash is presented in Table 1. The percentage loss on ignition (LOI) of Class F fly ash, which indicates the carbon content, is within the requirement of the standard.

The coarse aggregates (CA) used were graded 9.5-mm nominal maximum-sized crushed stone. The crushed stone had a bulk density of 1571 kg/m³, a specific gravity of 2.61, and 0.81% absorption. 9.5-mm crushed stone was used to ensure better mechanical performance, so that the differences in the mechanical properties between mixtures containing residuals and reference mixtures can be easily detected. The sand (S) used had a bulk density of 1706 kg/m³, a specific gravity of 2.66, 1.9% absorption, and a fineness modulus of 2.45.

The superplasticizer (SP) used in this research was an aqueous solution of a modified polycarboxylate conforming to the requirements of EN 934-2 [18]. The manufacturer recommends a dosage rate of 0.2–0.8% of the cement mass for medium workability.

2.2. Paper-mill residuals

The paper-mill residue used in this study was the primary sludge recovered from the first processing stage (primary clarifier) in a paper-mill. Tables 2 and 3 presented the properties of the residuals used in this research work. The scanning electron micrographs of the oven-dried primary residuals are showed in Fig. 1.

2.3. Developing the design matrix

The process control parameters are listed in Table 4. A total of 63 concrete mixtures were produced in this study which consist of Series 1 and 2 mixtures. Each series has different variables. Series 1 is non fly ash concrete, where its variables are water/cement ratio (*w/c*) and residual content (*R*). On the other hand, Series 2 concrete mixtures were developed with different fly ash content as replacement. A constant dosage of 0.4% superplasticizer was used to improve the workability

of Series 2 mixtures. The variables of Series 2 mixtures are paper-mill residual content (*R*) and ratio of fly ash to total cementitious material (*FA/CM*). The mixture proportions are presented in Table 5.

2.4. Preparation of concrete specimens and experimental programs

Prior to the mixing of concrete mixture, a process to deflocculate residual fibers is necessary to ensure the residual clump can be dispersed into individual fiber, and subsequently could be distributed evenly in the concrete mixtures.

Naik and Chun's method [5] was referred for the deflocculation of residuals. A high speed mixer was used to deflocculate or repulp the residual. Mechanical repulping was performed by immersing the fibrous residuals in room-temperature water until no further clump could be found.

Test specimens of concrete were made and cured according to the requirement of ASTM C 192 M-02 [17]. The concrete mixers used in this research were produced in the laboratory using a 110 kg capacity mechanical mixer.

The sequences of mixing and specimen preparation were done as follows:

The coarse aggregate and some of the mixing water were first added into the mixer. Then the mixer was allowed to start and stop after it turned a few revolutions. Then the fine aggregate was added, the mixer was allowed to start and stop after it turned a few revolutions. Then the cement, the rest of the water, and SP were added into the mixer. After all ingredients are in the mixer, the mixing continued for three minutes followed by a three minutes rest, followed by a two minutes final mixing. The consistency and workability of all the concrete mixtures were determined through slump tests. The slump tests were performed according to ASTM C 143-05a [17]. Compressive strength of 100-mm cube specimens were determined at 28 and 90 days in accordance with BS 1881: Part 116 [19].

3. Results and discussion

3.1. Development of mathematical model

In this study, the response surface models were developed by using Minitab software. Response surface methodology is a collection of mathematical and statistical techniques that are useful for modeling and analysis in applications where a response of interest is influenced by several variables.

If the response is well modeled by a linear function of the independent variables, then the approximating function is the first-order model, given by the following equation [16]:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \quad (1)$$

If there is curvature in the system, then a polynomial of higher degree must be used, such as the second-order model, given by Eq. 4.2 [20]:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 \quad (2)$$

where *y* is the response, β_0 the *y*-intercept (the value of *y* when $x_1 = x_2 = 0$), β_1 the coefficient of variable 1, x_1 the variable 1, β_2 the coefficient of variable 2, x_2 the variable 2, β_{12} the coefficient of interaction of variables 1 and 2, β_{11} the coefficient of square of variable 1, β_{22} the coefficient of square of variable 2.

Under some circumstances, a model involving only main effects and interactions may be appropriate to describe a response surface when the analysis of the results revealed no evidence of “pure quadratic” curvature in the response of interest or the design matrix originally used included the limits of the factor settings available to run the process.

3.2. Tests results

The slump decreased with the increase of paper mill residual content. The as-received residuals exhibited a high water absorption capability. Consequently, a higher amount of residuals in the mixture demanded more water to achieve a given slump. The workability of concrete containing paper-mill residual was improved by the addition of HRWRA. The compressive strength decreased as the unit weight of the concrete mixtures decreased. The unit weight of the concrete mixtures decreased with the increase of lighter paper-mill residual content as expected.

Table 1
Chemical composition of cement and Class F fly ash.

Oxide composition	Portland cement (PC) (%)	Class F fly ash (%)	Requirements for ASTM C 618 (%)
SiO ₂	21.54	62.5	
Fe ₂ O ₃	3.63	3.5	
Al ₂ O ₃	5.32	23.4	
CaO	63.33	1.8	
MgO	1.08	0.34	5.0 max
SO ₃	2.18	1.2	5.0 max
K ₂ O	–	0.95	
Na ₂ O	–	0.24	
LOI	2.5	5.61	6.0 max

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