

*Full Length Research Paper*

# Assessing the properties of freshly mixed concrete containing paper-mill residuals and class F fly ash

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**The disposal of wastewater treatment-plant solids collected from the paper-mill has become a crucial problem as the landfill space is limited. The use of paper-mill residuals in concrete formulations was investigated as an alternative to landfill disposal. Currently, there is still lack of information on the effect of fresh properties of residual concrete. Furthermore, the effect of class F fly ash on fresh properties of residual concrete is not yet discovered. The fresh properties of concrete containing paper-mill residuals and class F fly ash were investigated by different workability tests, fresh concrete unit weight and air content tests. The influence of the variables such as superplasticizer dosage, water/cementitious material ratio, residual content, and fly ash content on the workability tests was revealed in this study.**

**Key words:** Recycling, paper-mill, concrete, fly ash.

## INTRODUCTION

Wastewater treatment-plant solid residues from pulp and paper industry are those solid materials collected in the process of treating water used in the mill prior to its release into the environment. These wastewater treatment plant residuals consist predominantly of primary and secondary solids derived from primary and secondary treatment.

The solid residuals from pulp and paper industry are also one of the largest solid waste streams generated from the industries. For instance, the pulp and paper industry in United State generated 5.8 million dry tons of wastewater treatment-plant residuals in 1995, and only 2.8 million dry tons (49%) were managed in beneficial use applications, with the balance being disposed in a landfill (NCASI, 1999). The continuous increasing of wastewater treatment-plant residuals generated from paper industry has increased concern over the amount and quality of future landfill space. ACI 544.1R reported that the kraft pulp fibre that used as a processed natural fibre to reinforced concretes posses an ultimate tensile strength of 101,500 psi (700 MPa) (ACI Committee, 1996). The residual solids from wastewater

treatment-plant which still remains certain amount of cellulose fibres may have a potential to become an economical source for micro-fibre reinforcement of concrete.

Naik et al. (2003), mentioned that by using proper amounts of fibrous residuals in concrete formulation, water, and HRWRA, concrete mixtures containing the residuals were produced comparable to reference concrete mixtures (no residuals) in slump and compressive strength (Naik et al., 2003; Naik et al., 2005; Chun and Naik, 2005). With almost equivalent density, the concrete containing residuals can achieve higher splitting tensile and flexural strength than the reference concrete (Naik, 2005).

Fly ash, known as pulverized-fuel ash, is the ash precipitated electrostatically or mechanically from the exhaust gases of coal-fired power stations, is used in concrete for reasons including economics, improvements and reduction in temperature rise in fresh concrete, workability, and contribution to durability and strength in hardened concrete (Lane and Best, 1982; ACI Committee, 2003). It was found that adding to the paper mill residuals which are derived from recycled fibre source has shown no avail on the mechanical, durability and workability of concrete; however the including of fly ash in concrete showed a promising improvement in durability and long term strength

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**Table 1.** Chemical composition of PC and class F fly ash.

Oxide composition	PC, %	Class F fly Ash, %	Requirements for ASTM C 618
SiO <sub>2</sub>	21.54	62.5	
Fe <sub>2</sub> O <sub>3</sub>	3.63	3.5	
Al <sub>2</sub> O <sub>3</sub>	5.32	23.4	
CaO	63.33	1.8	
MgO	1.08	0.34	5.0 max
SO <sub>3</sub>	2.18	1.2	5.0 max
K <sub>2</sub> O	-	0.95	
Na <sub>2</sub> O	-	0.24	
Loss in ignition	2.5	5.61	6.0 max

development of paper-mill residual concrete (Mohammed and Fang, 2010).

The environmental and economic reasons are one of the important reasons for using paper mill residuals in concrete. According to Naik's study in United State (Naik et al., 2003), 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 U.S., there could be economic benefits of \$360 million for the concrete industry and \$30 million for the paper industry per year.

Since 1997, Naik has initiated several studies on the use of paper-mill residuals in concrete production (Chun and Naik, 2005; Naik et al., 2004). However, information on the properties of freshly mixed concrete containing paper-mill residuals is not enough and furthermore there is no research yet to study the rheological effect of fly ash as partial replacement of Portland cement in producing of concrete containing paper-mill residuals.

Primary residuals which are derived from the primary wastewater treatment in the paper-mill factory and class F fly ash were used to produce concrete in this study. The use of paper-mill residuals and class F fly ash in concrete formulation exhibited different rheological properties. This paper reports some quality characteristics of freshly mixed concrete which included paper-mill residuals, and class F fly ash as partial replacement of Portland cement.

## MATERIALS AND MIXTURE PROPORTIONS

### Portland cement, fly ash, coarse and fine aggregates

Portland cement (PC) Type I, which conforms to the requirement of ASTM C 150 – 04 (ASTM, 2004) was used to produce the concrete in this research work. The PC used has a specific gravity of 3.1. Class F fly ash, which conforms to the requirement of ASTM C 618 – 03 (ASTM, 2004) was used as partial replacement of PC. The fly ash was produced and received from Kapar Energy Ventures power plant in Malaysia. The specific gravity of Class F fly ash used is 2.04. 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 1,571 kg/m<sup>3</sup>, 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 1,706 kg/m<sup>3</sup>, 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 (British Standards Institution, 2001). The manufacturer recommends a dosage rate of 0.2 to 0.8% of the cement mass for medium workability.

### 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. Figure 1 shows scanning electron micrographs of the oven-dried primary residuals.

### Mixture proportions

The mixture proportions are presented in Table 4. A total of 77 concrete mixtures were produced in this study. The effects between water / cementitious material ratios (W/CM) with residual contents, and fly ash contents with residual contents on the compressive strength were first investigated by using two factors factorial experiment. The W/CM of this study was set at 0.37, 0.40 and 0.45. The residual content of 0, 0.25, 0.5, 0.75, 1.0, 1.5 or 2% (% of total cementitious material mass) was included in the concrete mixtures. The residual used in this research had undergone mechanical dewatering process in the paper mill, and assumed to be in saturated condition and would not further contribute to form the cement paste in the mixing. The w/cm is calculated from the mass of water included to react with the cementitious material to form cement paste. Superplasticizer dosage of 0, 0.2, 0.4 or 0.8% (L/100 kg of cementitious material) was applied in the concrete mixtures. The Class F fly ash contents ranged from 20 to 60% of the total cementitious material mass (20, 30, 40, 50 and 60% replacement) which were used as partial replacement of PC.

### Preparation of concrete specimens

Prior to the mixing of concrete mixture, deflocculating of residual fibres is necessary to ensure the residual clumps can be dispersed into individual fibre, and subsequently distributed evenly in the concrete mixtures.

Naik and Chun's method (2003) was employed 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 clumps were observed.

Test specimens of concrete were made and cured according to the requirement of ASTM C 192M – 02 (ASTM, 2004). The mixing of concrete started with adding the coarse aggregate and some of the mixing water into the mixer. The mixer was then 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

**Table 2.** Physical properties of paper-mill residuals.

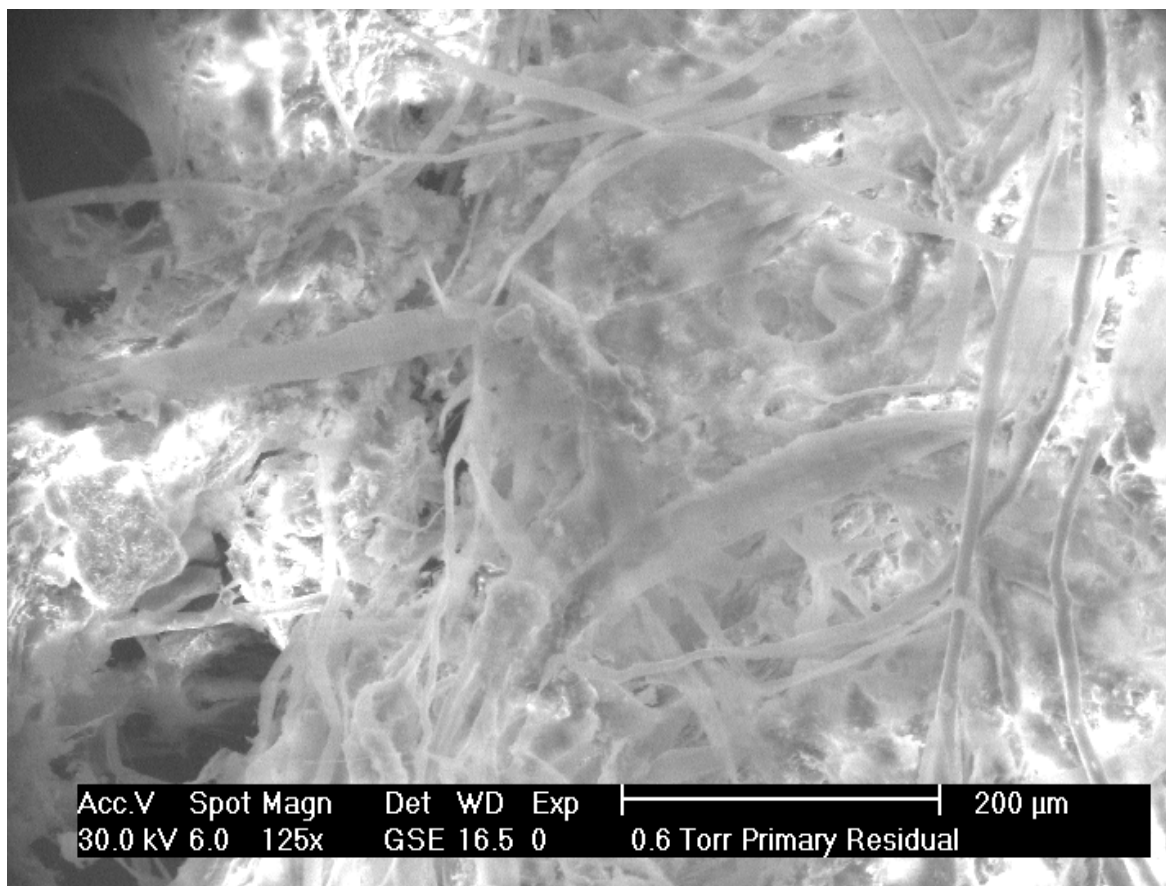
Type of residual	Type of mill	Fibre origin	Moisture content (%) <sup>a</sup>	Apparent specific gravity	Loss on Ignition at 590°C (%) <sup>a</sup>	Wood- fibre content (%) <sup>b</sup>
Primary	Paper	Recycled	225	1.661	46	36

<sup>a</sup>Percent of oven-dried (105 °C) mass of residuals; <sup>b</sup> Wood Fibre Content (%) = 1.083 \* LOI at 590°C - 14.1 (Naik et al., 2003).

**Table 3.** Oxides composition of ash left after ignition of dried residuals at 1,000°C.

Element	% by mass a
LOI at 1,000°C	53.450
MgO	2.140
Al <sub>2</sub> O <sub>3</sub>	8.620
SiO <sub>2</sub>	15.100
K <sub>2</sub> O	0.290
CaO	17.400
TiO <sub>2</sub>	0.707
Fe <sub>2</sub> O <sub>3</sub>	1.660
Total	99.985

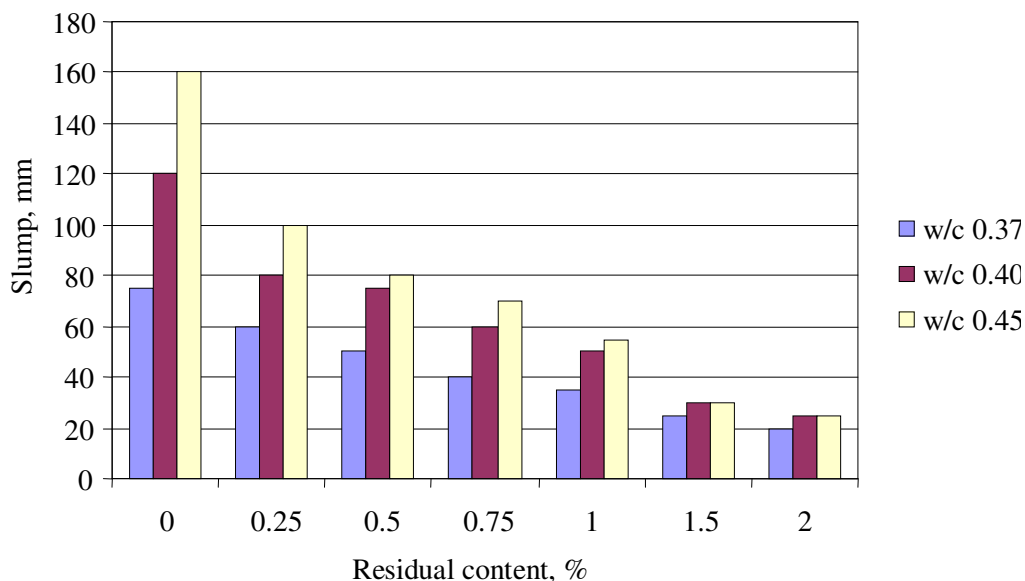
<sup>a</sup>Percent of oven-dried (105°C) mass of residuals.

**Figure 1.** Scanning electron micrographs of oven-dried primary residuals.

**Table 4.** Mixture proportions.

Total mixtures	CM:S:CA:W	Residual content, % or kg/100 kg of cement <sup>a</sup>	SP, % or L/100 kg of cement	Fly ash replacement, %
28	1:0.767:1.275:0.37	0 - 2	0 - 0.8	0
35	1:0.767:1.275:0.37	0 - 2	0.4	20 - 60
7	1:0.889:1.378:0.4	0 - 2	0	0
7	1:1.093:1.55:0.45	0 - 2	0	0

<sup>a</sup>As-received moist residuals were used. The quantities of residuals shown are on oven-dried basis.

**Figure 2.** Effect of residual content on slump.

were added into the mixer. After all ingredients were in the mixer, the mixing continued for three minutes followed by a three minutes rest, and a two minutes final mixing.

The fresh concrete properties were further determined and 100-mm cube specimens were cast to determine the 28 days compressive strength.

#### Experimental program and test procedures

Immediately after the completion of mixing, the fresh concrete was tested on the workability properties, unit weight and air content. The types of workability test and its conformity standard are stated as:

1. Slump test – ASTM C 143-03 (ASTM, 2004)
2. Compacting factor test – BS 1881: Part 103:1993 (British Standard Institution, 1993).
3. Vebe test – BS EN 12350-3:2000 (British Standards Institution, 2000).

The unit weight and air content tests were performed simultaneously with the workability tests and the test procedures are conformed to ASTM C 138M – 01a (ASTM, 2004).

Compressive strength of the 100-mm cube specimens was determined at 28 days in accordance with BS 1881-116:1983 (British Standard Institution, 1983).

## RESULTS AND DISCUSSIONS

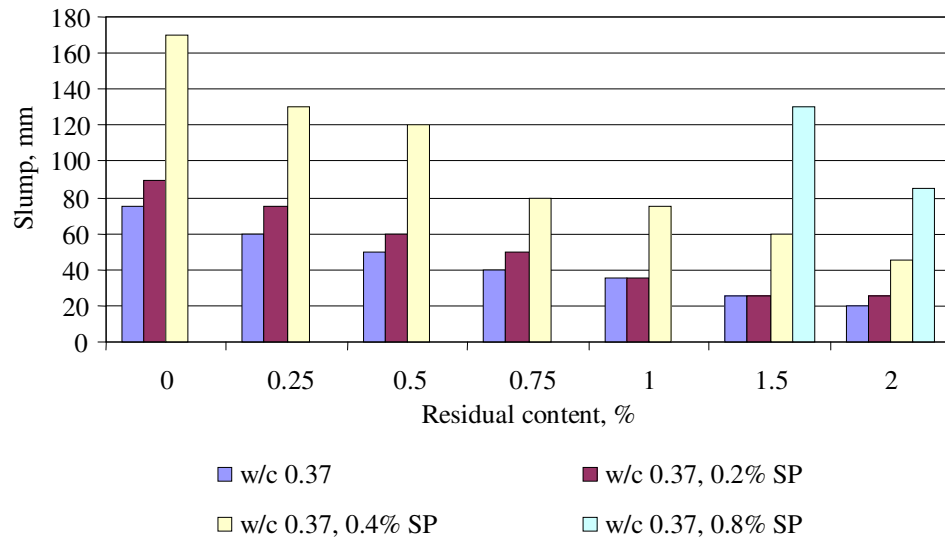
### Compressive strength

From the experimental results, the 28 days compressive strength of the concrete decreased when residual content and fly ash content increased in the mixtures. Due to the mixture proportion, the compressive strength decreased when W/CM ratio increased. The effect of superplasticizer dosage on the compressive strength of concrete was found to be insignificant.

### Slump test

#### *Effect of paper-mill residual on slump test*

The effect of residual content on the slump is shown in Figure 2. The slump values reduced when residuals contents increased. The results shown are in good agreement with the observation of Tarun (Naik et al., 2003; Chun and Naik, 2005), where the paper-mill



**Figure 3.** Effect of superplasticizer dosage on slump test.

residuals could increase water demand of concrete for a given slump. The as-received residuals exhibit a high water absorption capability. In consequence, when higher amount of residuals is included in the mixture, it requires more water to achieve a given slump.

#### ***Effect of superplasticizer dosage on slump test***

Figure 3 presents the slump results of residual concrete with different superplasticizer dosage. Slight or no improvement in the slump values were observed between the mixtures with no superplasticizer and 0.2% superplasticizer at each of their respective residual content. The concrete mixture containing residuals has improved their workability through the incorporation of 0.4% superplasticizer, and has achieved workability range from medium to high depending on the residual content. The mixture with 0.8% of superplasticizer recorded collapse slump for residuals content from 0 to 1% of cement mass, and a relatively high workability could be observed in the mixtures of 1.5 and 2% residual content.

With proper combination of residuals and superplasticizer, workability of concrete can be adjusted. The workability of concrete containing paper-mill residuals was improved by using superplasticizer. Higher dosage of superplasticizer would result in higher workability. However, it should also be noticed that bleeding and segregation could be observed when 0.8% superplasticizer is introduced into the concrete mixtures containing of 0 to 0.5% of residual contents.

Apparently, slump test is not suitable to determine the workability of concrete with 0.8% superplasticizer and containing 0, 0.25, 0.5, 0.75 and 1% residual contents

since the slump test is not applicable when slump value higher than 230-mm is limited in ASTM C 143–03 [ASTM, 2004].

#### ***Effect of fly ash content on slump test***

Figure 4 presents the slump results of residual concrete with different fly ash content. The slump values reduced when fly ash contents increased. A very low slump, 5-mm was observed in the mixture of 60% of fly ash replacement and 2% residuals content. The slump test might not be suitable to determine the workability of concrete when the slump is less than 10-mm.

In this investigation, the workability of concrete was governed by the fly ash content and also the residuals content. Increasing of fly ash content and residuals content would lead to increase in water demand of fresh concrete to achieve a given slump. The replacement of cement with fly ash is known to contribute to a reduction in water demand and an improvement of workability due to the smaller size and the essentially spherical form of the fly ash particles (Berry and Malhotra, 1980). However, the literature does contain some contrary data. According to CUR Report 144 (Bijen and Selst, 1992), an amount of fly ash in excess of that required to cover the surface of the cement particles would confer no further benefit with respect to water demand. The reduction in water demand becomes larger with an increase in the fly ash content only to about 20%.

Brink and Halstead (1956) have reported that some fly ashes reduced the water requirement of test mortars, whereas others (generally of higher carbon content) showed increased water requirement above that of control mortars. Welsh and Burton (1958) reported loss of

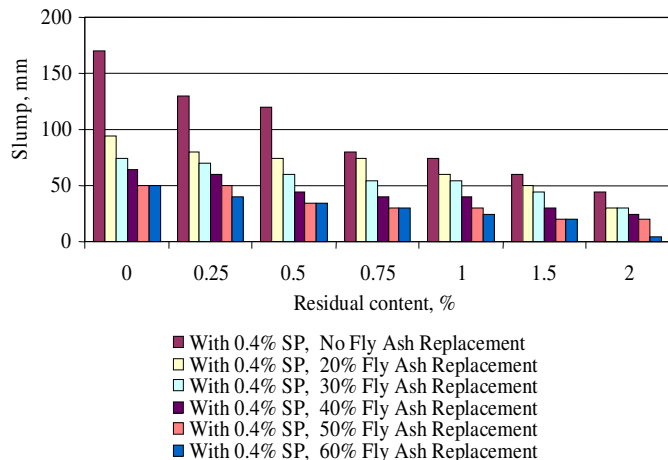


Figure 4. Effect of fly ash content on slump test.

slump and flow for concretes made with some Australian fly ashes used to partially replace cement, when water content was maintained constant. Reshi (1973) has reported that experience with a number of Indian fly ashes showed that all those examined increased the water requirement of concrete.

The carbon content of the class F fly ash used in this study was 5.61% based on LOI testing. Although the carbon content was still within the 6% allowed by ASTM, the relatively high carbon content might have led to the adverse effect on workability.

### Relationship between slump, compacting factor and Vebe time

The relation between slump, compacting factor and Vebe time is presented in Figure 5. Generally, the workability was found to be reduced when residual content and fly ash content increased in the concrete mixtures, and the workability could be improved by increasing of W/CM ratio and superplasticizer dosage. Similar trend of workability could be found in the slump test results, as well as compacting factors and Vebe times. The range of slump in this study was within 5 to 170-mm. ASTM C 143–03 (ASTM, 2004) stated the limitation of slump test, which was only applicable for slump in the range of 15-mm to 230-mm. It can be seen that at low workability, most of the slump recorded the same value while Vebe test recoded a wider range of Vebe time.

Through the observation of relationship, Vebe test seems to be more sensitive in the low workability condition than slump test and compacting factor test. However, slump test and compacting factor test provide better indication of workability at high workability. It is due to the vibration time of Vebe tests of high workability mixtures are relatively close between each other since there are not much effort required in the compaction. BS

Table 5. Analysis of variance of slump tests.

Slump tests				
Terms	W/CM	Residual	SP	Fly ash
F	8.94	31.23	35.53	23.41
P	0.0004	<0.0001	<0.0001	<0.0001
S		13.738		
R <sup>2</sup>		86.53%		

Table 6. Analysis of variance of compacting factor tests.

Compacting factor tests				
Terms	W/CM	Residual	SP	Fly ash
F	4.07	42.28	28.04	12.68
P	0.022	<0.0001	<0.0001	<0.0001
S		0.034		
R <sup>2</sup>		86.53%		

Table 7. Analysis of variance of Vebe tests.

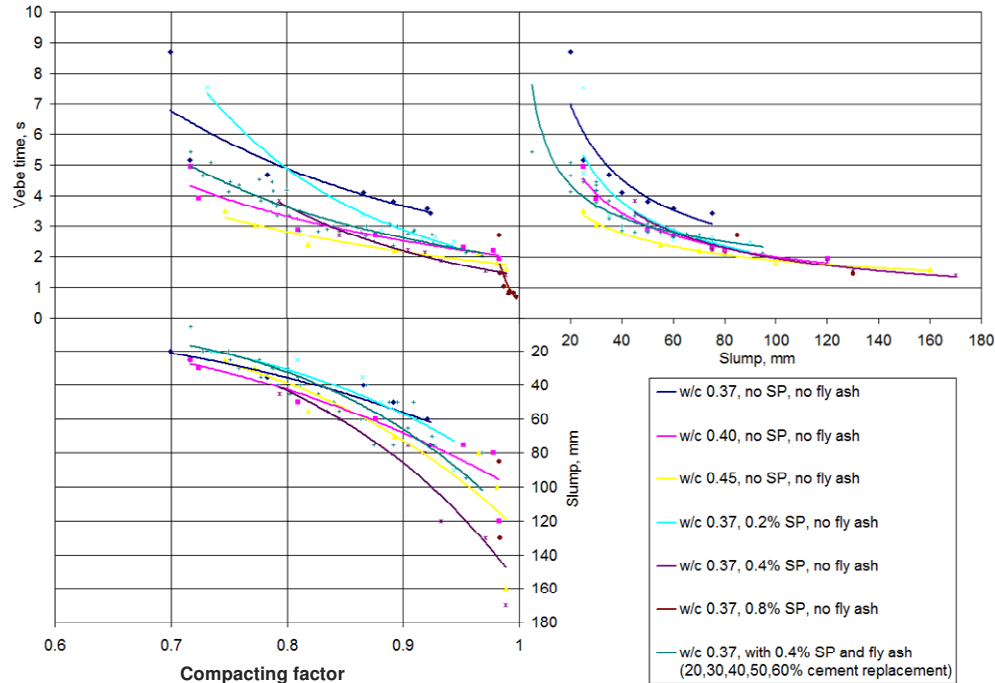
Vebe tests			
W/CM	Residual	SP	Fly ash
44.48	40.27	71.26	13.04
0.022	<0.0001	<0.0001	<0.0001
		0.034	
		86.53%	

EN12350-3 (British Standard Institution, 2000) also stated the Vebe time should be in the range of 5 to 30 s.

The compacting factors recorded in this study ranged around 0.7 to 1. However, BS 1881: Part 103 :1993 (British Standard Institution, 1993) stated that the normal range of compacting factor test lies between 0.8 - 0.92. The sensitivity of the compacting factor is reduced outside the normal range of workability and is generally unsatisfactory for compacting factor greater than 0.92. Thus, Vebe test provides better indication at low workability, while slump test provides better indication at high workability as compared to compacting factor test.

From the result, slump test and compacting factor test seem to be a more suitable testing for the concrete containing residuals, except for the mixture with high residual content (2%) and high fly ash portion mixture (60% fly ash replacement). On the other hand, Vebe test is merely suitable for testing with low W/CM ratio, high fly ash content, and low superplasticizer mixture.

Tables 5 - 7 show the analysis of variance for slump, compacting factor, and Vebe tests. Within the range of the test variables, the analysis of variance showed that



**Figure 5.** Relation between workability tests for different mixture category.

the factors that influence slump according to descending order are:

1. Superplasticizer dosage
2. Residual content
3. Fly ash content
4. W/CM

The result leads to the observation that slump test was more sensitive to the superplasticizer dosages while W/CM ratio has relatively less influence on the slump. When slump test was performed, the slump occurred when the concrete specimen sheared its self-weight. As the superplasticizer dosage increases, the dispersion of cement particles would reduce the friction and the shear of concrete specimen, eventually causing a great influence on the slump.

The analysis of variance showed that the factors that influence compacting factor according to descending order are:

1. Residual content.
2. Superplasticizer dosage.
3. Fly ash content.
4. W/CM ratio.

Note that the residual content plays a more important role in the compacting factor than the slump and Vebe test. When higher residual content was introduced in the mixtures, the mixtures became more cohesive, and

subsequently affected the free-fall process of the compacting factor test and the mass of partially compacted concrete collected. Hence, the influence of residual content on compacting factor is more than slump test and Vebe test.

The analysis of variance showed that the factors that influence Vebe time according to descending order are:

1. Superplasticizer dosage.
2. W/CM ratio.
3. Residual content.
4. Fly ash content.

Apparently, the superplasticizer dosage is sensitive to the vibration of Vebe test. Besides, the effect of residual content and fly ash content to Vebe time is not as significant as slump test and compacting factor test. It is due to the vibration compaction of Vebe test that could compact the mixtures containing paper-mill residuals and fly ash more easily than the rod tamping method of slump test and compacting factor test.

### **Unit weight**

The fresh concrete unit weights of concrete mixtures are presented in Figure 6. The unit weight of concrete mixtures decrease with increase of residuals content. There was no significant difference observed between the unit weights of the reference concrete mixtures with

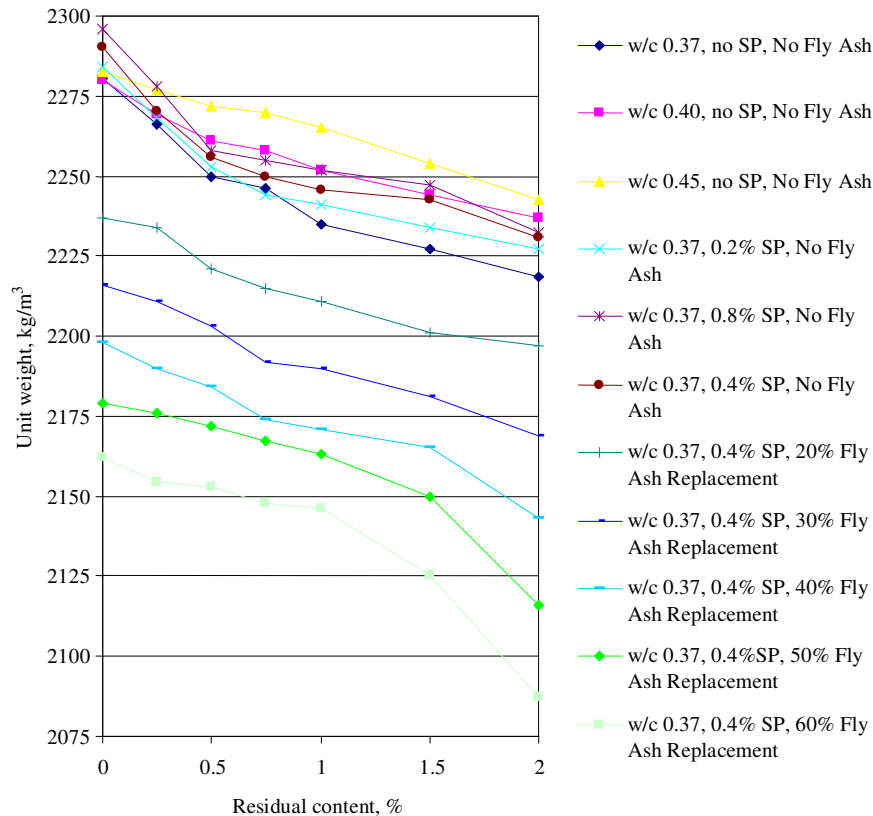


Figure 6. Unit weight versus paper-mill residual content.

different W/CM ratio. However, when higher residual content was introduced in the mixtures, the unit weight also increased as W/CM ratio increased. Higher dosage of superplasticizer could produce concrete mixtures with slightly higher unit weight. The replacements of cement with fly ash have resulted in comparative greater difference in the unit weight of concrete mixtures. The unit weights of mixtures containing fly ash decreased with higher fly ash content in the concrete mixtures.

The paper-mill residual, which has a specific gravity of 1.661, is a light weight ingredient comparing to the aggregates and cementitious material used. Thus, when more residuals are included in the mixtures, more volume in the concrete would be occupied by the residuals and it would reduce the overall unit weight.

The increase of unit weight when W/CM ratio increased in the concrete mixtures which have the same residual content was due to the decreasing of air content when higher W/CM ratio was introduced in the mixtures.

The superplasticizer improved the concrete mixture workability and orientated the paste more compactly. Hence, higher dosage of superplasticizer probably would slightly increase the unit weight of the concrete mixtures containing the same residual content.

Fly ash has a lower specific gravity ( $2.04 \text{ kg/m}^3$ ) than Portland cement ( $3.1 \text{ kg/m}^3$ ). Substitution of fly ash for

an equal weight of Portland cement therefore increased the paste volume in the concrete and reduced the density of concrete. Hence increasing fly ash content in concrete mixture would reduce the overall concrete unit weight.

### Air content

The air contents of concrete mixtures are presented in Figure 7. The air contents of concrete mixtures were affected by residual content, W/CM ratio, dosage of superplasticizer, and fly ash content.

Higher residuals content in the concrete mixtures resulted in higher air content. The air contents decreased as W/CM ratio increased and higher superplasticizer dosage resulted in lower air content. Generally, higher fly ash content showed lower air content in the concrete mixtures, exceptions were found in mixture of 50% cement/fly ash replacement with 2% of cement mass of residuals, and 60% cement /fly ash replacement with 1.5 and 2% of cement mass of residuals.

Since paper-mill residuals exhibited high water absorption characteristic, higher residual content included into the concrete mixture would absorb more water which is used to form cement paste. Eventually, the workability of concrete mixtures would reduce and more pores would



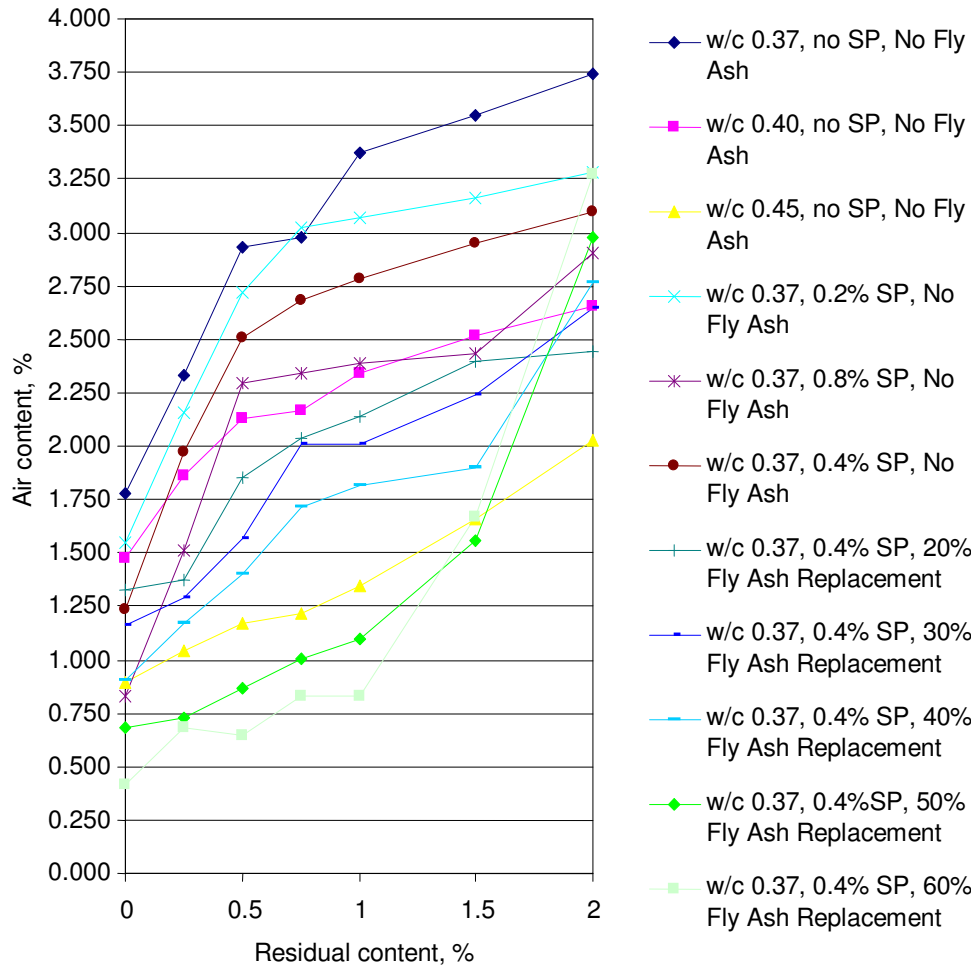


Figure 7. Air content versus paper-mill residual content.

exist in the concrete mixture which contributes to higher air content.

The concrete mixtures of higher W/CM ratio would have more water content for cementitious material to form cement paste rather than been absorbed by the fibrous residuals and eventually create pores around the residuals in the mixtures. Hence, when W/CM ratio increased, the air content of concrete mixtures decreased. The results also indicate that by adding superplasticizer in the mixtures, it improved the workability of concrete and orientated the mixture more compactly, leaving lesser space for air content.

The effect of fly ash on the air content of concrete mixtures generally agrees with the previous researchers (Lane and Best, 1982; ACI Committee, 2003; Lane, 1983). Higher fly ash content in concrete mixture could decrease the air content since fly ash is more effective to fill the voids in the concrete mixtures, which are due to its fineness and shape. The exceptions found in the high cement replacement might be caused by the reducing of workability of concrete.

## Conclusions

Based on the results presented, the following conclusions can be drawn:

(i) Generally, higher residual content and fly ash content in the concrete mixtures would increase the water demand of concrete for a given slump, thus, decreasing the workability of fresh concrete. The workability of concrete containing paper-mill residuals and fly ash content could be adjusted and improved by using proper amount of superplasticizer. The Class F fly ash decreased the workability of concrete due to its high percentage of fly ash replacement in mixture proportion and high carbon content which increases the water demand.

(ii) The slump test and compacting factor test were the most suitable testing for the workability of concrete containing paper-mill residuals as compared to Vebe test. However, the influence of the variables such as superplasticizer dosage, W/CM ratio, residual content,

and fly ash content were varied for each respective type of tests.

(iii) The unit weight of fresh concrete decreased with increasing of residuals content and fly ash content. Including superplasticizer in the concrete mixtures resulted in slightly higher unit weight compared with mixtures without superplasticizer content.

(iv) Higher residuals content in the concrete mixtures resulted in higher air content. Superplasticizer and Class F fly ash could reduce the air content of the concrete mixtures containing paper-mill residuals.

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