Evaluation of Potential use of Waste Powder Paint (WPP) in Cement Grout and Mortar

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Research

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ABSTRACT

A wide range of polymeric admixtures is used in cement-based products to improve the fresh and hardened properties. The waste powder paint (WPP) that is basically made of different types of polymers is available in abundant quantity from auto and furniture industries' discharge. This material is usually dumped into the landfills. To help sustainability, it is vital to explore potential use of WPP in other industries. The use of WPP in cement-based products such as grout and mortar was evaluated by analyzing their fresh and hardened properties. This study was limited to the testing of WPP as a cement replacement. Preliminary results with WPP showed reduction in heat of hydration, reduction in compressive strength at early ages, continuous strength development with WPP in the presence of moisture, significant expansion opposed to the shrinkage properties observed with cement, and altered absorption and leaching mechanisms. It is demonstrated from the limited investigations conducted during this project that WPP can be effectively used in cement-based products where expansive and water repellency properties are required. However, additional research is needed to evaluate WPP material properties and their influence on cement hydration, microstructure of cement-based product with WPP, fresh and hardened properties, durability properties, and mixing, transport, and placement methods. The outcome of the research could allow WPP to be used in the following civil engineering applications.

1. Grouting of connection between precast elements when there is a need of non-shrink/expansive filling material.
2. Filling voids with restricted access by pumping or with gravity flow.
3. Underpinning of old structure foundations, when there is a need for less dense cementitious material with expansive nature. The compressive strength is not a critical parameter for such applications.
4. Filling masonry wall cavities with expansive grout or mortar.
5. Making masonry blocks and paving/cladding stones. However, this requires further investigation into the WPP chemicals that affect water repellency, absorption, and leaching due to their continuous exposure to moisture.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

1 INTRODUCTION

Powder paint is used in many industries including automotive and furniture manufacturing. When the powder paint is sprayed on to the components, a significant amount is wasted. As per a survey of six companies carried out by the Green Manufacturing Initiative (GMI) at Western Michigan University, waste powder paint (WPP) amounts to 1.5 million pounds per year. In general, WPP needs to be discarded into landfills. There is an interest to use WPP in other industries as a recycled material. However, the degradation of re-processed polymer and the high volume of waste exceeding the capacity of the outlets challenge the recycling process. Due to these recycling challenges and the significance of the monetary expenses involved in using WPP as a landfill material, there is an interest of exploring the use of WPP in other industries, without further processing, to promote sustainability. One such potential application is the use of WPP in masonry or concrete products. To promote such applications, fresh and hardened properties of mortar and concrete with WPP need to be evaluated.

1.1 PROBLEM STATEMENT AND BACKGROUND

Powder coating industry is facing difficulties in recycling the waste powder paint (WPP) to be used as a useful product (Fu et.al. 2011). These difficulties arise due to lean manufacturing practices, in which the waste from different color powder paints is accumulated along with mixing of materials from different powder technologies. In general, the WPP is a mixture of Epoxy-Polyester Hybrid, Polyester Triglycidyl Isocyanaurate (TGIC), Urethanae-Polyesters, Polyesters-Hydroxl, and Epoxy based materials (GMI 2011).

This cement-based industry has a proven track record of using fibers, polymers and polymer-modified particles, and other additives such as silica fume to improve concrete, mortar, and masonry properties. Enhanced strength and durability properties of cement-based materials play a significant role in reducing life-cycle cost (Mehta 1999). The use of polymers to improve the properties of cement-based products such as concrete, mortar, and grout is an emerging field of interest in civil and construction engineering (Gemert et.al. 2005). Even though there are no records of using WPP in cement based products, powdered polymers have been used to develop polymer-modified mortar and concrete (Afridi et al. 2003; Gemert et al. 2005). The use of additives in concrete has proven to be increasing compressive strength, abrasion resistance, and

bonding between reinforcement and cement matrix, and reducing drying shrinkage, and permeability (Xu and Chung 1999).

Afridi et al. (2003) tested commercially available powdered and liquid cement modifiers to evaluate the coalescence of polymer particles (*continuous polymer films formation)* in powdered and liquid polymer-modified mortars. They are *polyvinyl acetate-vinyl carboxylate (VA/Veo Va) type and polyethylene-vinyl acetate (EVA) type* powdered modifiers *and styrene-butadene rubber (SBR) latex type* liquid modifiers. Both of these modifiers have proven to be forming continuous polymer films in making a monolithic polymer-cement matrix when added to the cement-based mixture. This polymer film seems to be creating elastic interconnections between aggregates and cement hydrates by filling or reinforcing the capillaries and cavities (Afridi et al. 2003, Ohama et al. 1984). The final polymer film may appear as mesh-like, rugged, dense or fibrous with fine or rough surfaces (Afridi, et.al. 2003). The polymer formation can occur in different stages of cement hydration process depending on the polymer-cement ratio. The film formation and cement hydration process depends on the curing temperature and duration. The polymer film can partly or completely envelop a cement grain, which results in a retardation or even a complete stop of a hydration of the cement grain. On the other hand the polymer film can intermingle with cement hydrates (Gemert, et.al. 2005).

The polymer-to-cement ratio by weight of 10% - 15% has been found optimal in forming a coherent polymer film and developing greater flexural capacities (Afridi et al. 2003; Gemert et al. 2005). There are two potential options for the powdered polymers to be mixed with the cement-based product. The first option is to mix in powder form with cement (dry mixing). The second option is to dissolve the powdered polymers in a proper solvent and to mix in liquid form (Afridi, et.al. 2003).

Since there are no records of using WPP in cement based products, physical and chemical properties of WPP as well as fresh and hardened properties of mortar and concrete with WPP need to be evaluated. The composition of the WPP, which is intended to be used in this project, is a mixture of 69% Epoxy-Polyester Hybrid, 25% Polyester Triglycidyl Isocyanaurate (TGIC), and a mixture of Urethanae-Polyesters, Polyesters-Hydroxl, and Epoxy based materials (Figure 1) (GMI 2011).

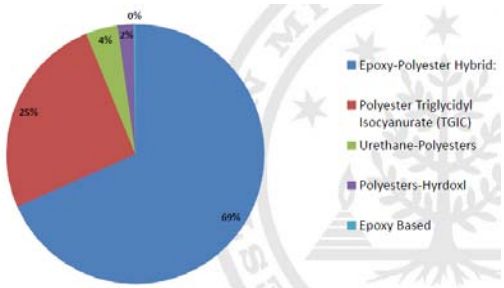


Figure 1. Composition of powder paint *(Source: GMI 2011)*

The temperature at which the powder paints changes it phases is of concern. Powder paint has three different transition temperature ranges. For an example, the ranges of glass transition temperature, rubber-elastic state temperature, and melting temperature of automotive powder clear coats are 30 - 60 ^{0}C, 50 - 100 ^{0}C, and 90 - 190 ^{0}C, respectively. The chemical cross-linking of a thermally curable powder coating starts to be noticeable within or above the melting temperature range (Graewe and Rettig 2002). A typical cement mixture may reach 60 - 70 ^{0}C (140 - 160 ^{0}F) during the hydration process (PCA 2003). In order to understand the phase transition of WPP during cement hydration, thermal behavior of WPP needs to be evaluated.

Waste powder paint typically contains the smaller particles compared to the virgin powder paint because the larger particles are less likely to be blown away from the work target due to the higher momentum (Fu et.al. 2011). This is evident from the particle size distribution analysis of used (waste) and virgin powder paint samples which show a mean value of 37 μm and 54 μm respectively (Figure 2) (GMI 2011). On the other hand, approximately 95% of cement particles are smaller than 45 μm with the average particle size of 15 μm (PCA 2003). Hence, the WPP and cement have comparable particle sizes.

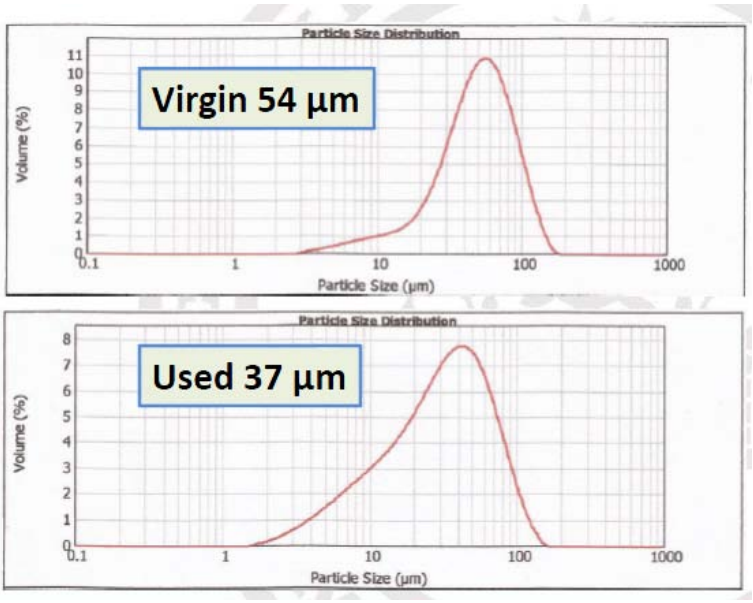


Figure 2. Particle size distribution of virgin and used (waste) powder paint (Source: GMI 2011)

The result of WPP solubility tests performed by a GMC research group is shown in Figure 3. However, there is no interpretation of the results present in the original document, provided by GMI research group, to identify the solvents for dissolving WPP.

#	Solvent	Chem. Formula	δ (cal/cm^3)$^{1/2}$	µ(D)
1	ethanol	C_2H_5OH	12.92	1.7
2	isopropanol	C_3H_8O	11.9	1.6
3	acetone	C_3H_6O	9.9	2.9
4	toluene	C_7H_8	8.9	0.4
5	chloroform	$CHCl_3$	9.21	1.0
6	N-methysl-2-pyrrolidone (NMP)	C_5H_9NO	11.1	3.8
7	dimethylformamide (DMF)	C_3H_7NO	12.14	3.8
8	hexane	C_6H_{14}	7.3	0.0

Figure 3. Solubility test results (Source: GMI 2011)

1.2 OBJECTIVE OF THE RESEARCH

The objective of the short-and long-term research is to investigate the influence and interaction of various parameters and constituents of WPP, cements, supplementary cementitious material (SCM), and chemical admixtures on fresh, hardened, and durability properties of cement-based products. The overall objective is presented in Figure 4. Table 1 provides detailed breakdown of parameters or properties of concern associated with materials, operations, reactions, and final products.

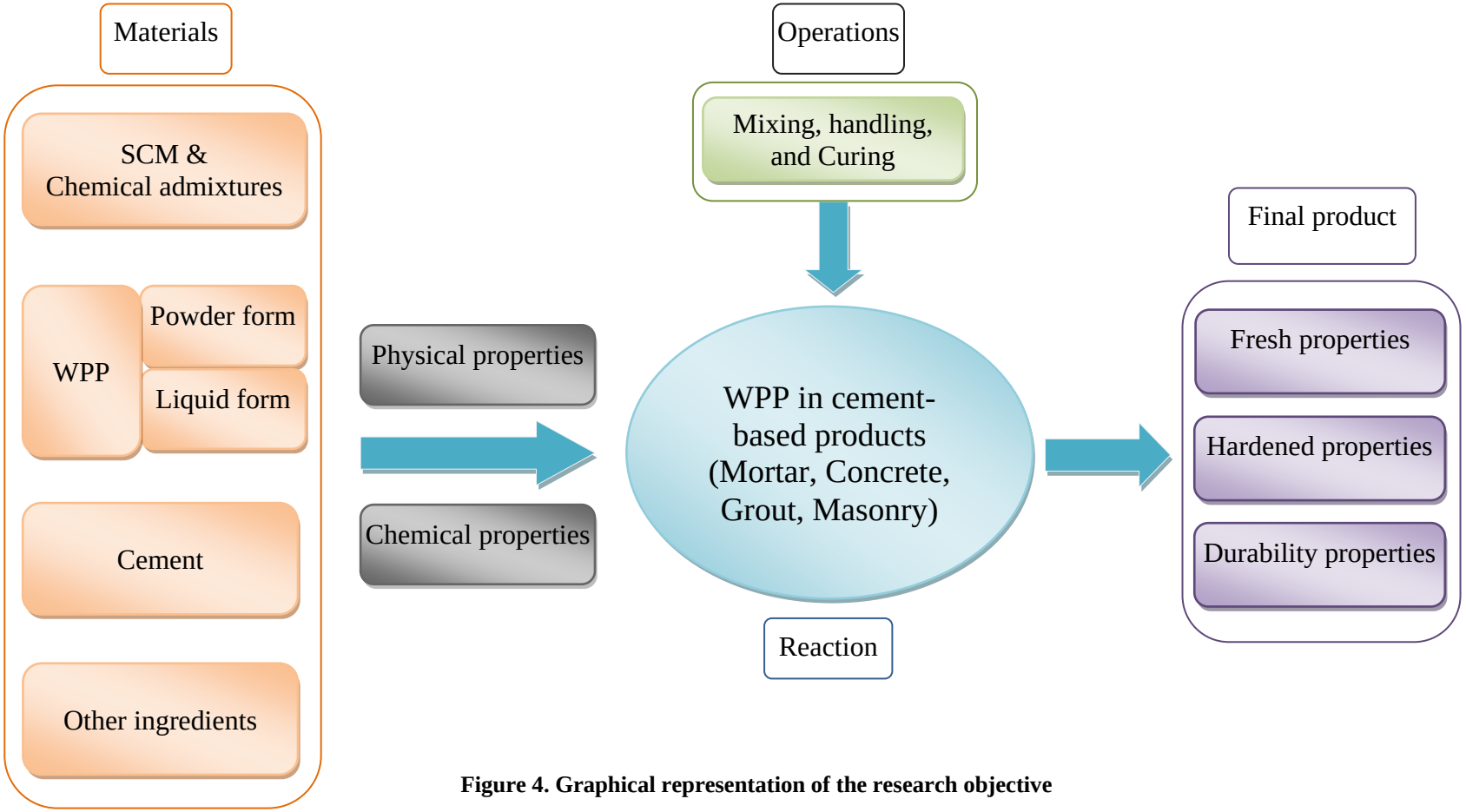


Figure 4. Graphical representation of the research objective

Table 1. Detailed Breakdown of Parameters or Properties associated with Materials, Operations, Reactions, and Final Products

Materials	Mixing , Handling, and Curing	Reaction (WPP + Cement mixture)	Final product
Waste powder paint Physical properties (powder form) • Particle size distribution • Particle shape • Surface properties Physical properties (liquid form) • Surface tension • Viscosity Chemical properties • Surfactants and other additives **Cement** • Chemical and physical properties **Aggregates** • Fine aggregate • Coarse aggregate • Chemical and physical properties **Supplementary cementitious materials and chemical admixtures** • Physical and chemical properties	Mixture proportions / Mix design Mixing equipment Transportation equipment Placement methods and equipment Curing methods/requirements • wet curing • dry curing • curing duration • curing temperature	WPP particle dispersion and distribution Hydration process and chemical reaction of the cement mixture and WPP Reaction of WPP as a polymer and comparison to • Admixture polymers • Polymer-modified concrete WPP as an additive/filler material in the cement mixture Optimum temperature requirements Effect of surfactants	**Fresh properties** • flowability/workability • setting time • heat of hydration • volume change • air content • unit weight **Hardened properties** • rate of strength gain • flexural strength • tensile strength • unit weight • porosity/permeability • modulus of elasticity • bond strength • resistivity • shrinkage • creep • soundness • void parameters **Durability properties** • freeze/thaw resistance • chemical resistance • volume stability • thermal properties • leaching behavior • absorption • permeability • Crack bridging behavior • Water repellency • Abrasion resistance

2 COMPOSITION OF CEMENT AND WASTE POWDER PAINT

2.1 OVERVIEW

Waste paint is a potentially valuable resource, which is currently being disposed at a financial and environmental cost. This valuable resource is made up of copious amount of fine particles, usually in the range of 35 - 55 μm (GMI 2011). The majority of powder paints include surfactants, titanium dioxide (transparent small particles which due to its small size, reflect the light and appears to be white) and thickeners (Almesfer et al. 2012). The influence of waste powder paint (WPP) on fresh and hardened properties of cement-based products has not been studied. Understanding of chemical composition of WPP is important to explain the changes in fresh and hardened properties when observed through experimental data. With that objective the information in this section is compiled from material datasheets and literature.

2.2 SURFACTANTS

Surfactants are used in power paint. Understanding of commonly used surfactants and their characteristics will help explaining the potential interaction of these chemicals with cement. An in-depth study of surfactants used in power paint is not the focus of the study. Hence, common characteristics of surfactants are reviewed. As the name implies, surface-active agents or surfactants are materials which are active at surfaces, or interfaces between two physical phases. Surfactants can be divided into ionic and nonionic types. Ionic surfactants can be further divided into anionic, cationic and amphoteric (Schmitt 1992). Anionic surfactants are also known as alkyl benzene sulfonates. The surfactant molecule consists of a polar and non-polar hydrocarbon chain. The polar is referred to as the ‘head’ while the non-polar chain is referred to as the ‘tail.’ The non-polar chain is hydrophobic. Hence, when the molecules get in contact with water, the non-polar chains tend to move away from the water molecules while the heads, hydrophilics, are present outside for interaction with water. The relative sizes or chain length and chemical natures of the hydrophilic and hydrophobic groups determine the characteristics of the surfactant molecule (Bronze 1999).

Based on the amount of surfactant usage, the conditions shown in Figure 5 may occur. The first column represents various concentrations of surfactants (from top to bottom) very low concentrations, low concentrations, at the Critical Micelle Concentration (CMC) and above the

CMC. As shown in the second column, the hydrophobic 'tail' is moved towards the hydrophobic surface while the hydrophilic 'head' is moved towards hydrophilic surface. At or above CMC, a micelle, a ball type structure is formed. In this particular structure, non-polar hydrocarbon chains or 'tails' are clustered into the center of the ball while hydrophilic heads are directed outward to interact with water molecules.

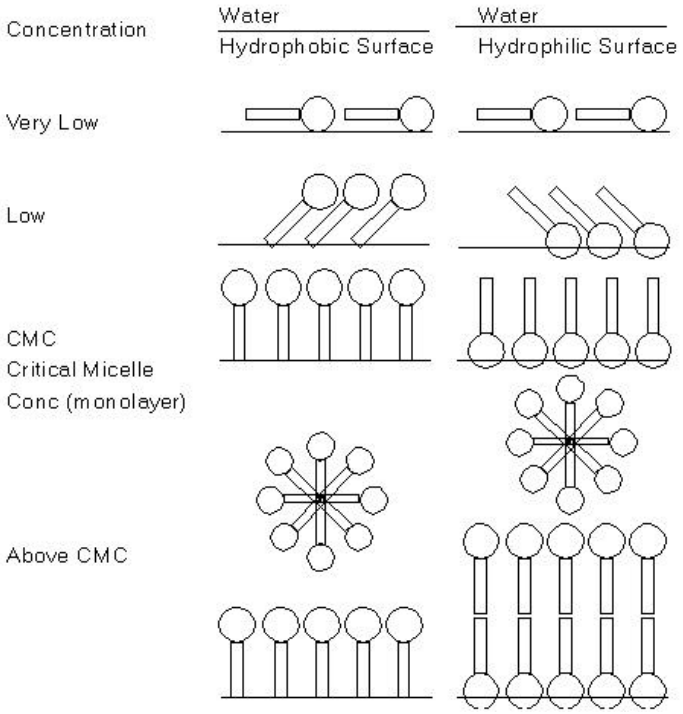


Figure 5. Graphical representation of anionic surfactant response against its concentration (Bronze 1999)

2.3 CEMENT CHEMISTRY

From a molecular perspective, cement is a paste of calcium silicate hydrates polymerized into a densely cross linked matrix. Portland cement raw material is composed of about 63% calcium oxide, 20% silica, 6% alumina, 3% iron (III) oxide, and small amounts of other matter including impurities (Hewlett 2004). The hydration process is complex and is the subject of extensive research.

Hydration is a string of chemical reactions between cement and water in which the cement-water paste sets and hardens. Once cement comes in contact with water, hydration process begins. Hydration process starts with the dissolution of 'C_3S'. H^+ in the water and react with the oxygen ions on the surface of the 'C_3S' lattice and form hydroxide ions, which combine with Ca^{2+} to form $Ca(OH)_2$:

$$O^{2-}\ (lattice) + H^{+}(aq) \rightarrow OH^{-}(aq) \quad (1)$$

$$2OH^{-}(aq) + Ca^{2+}(aq) \rightarrow Ca(OH)_2\ (aq) \quad (2)$$

Simultaneously, silicate material from the 'C_3S' lattice surface is introduced into the liquid phase:

(3)

The dissolved components coalesce to form the calcium silicate hydrate 'CSH' gel, an amorphous solid solution composed of $Ca(OH)_2$ and a calcium silicate hydrate of low Ca:Si ratio. The hydration can be shown as the example below:

$$2(3CaO.SiO_2) + 6H_2O(l) \rightarrow 3CaO.2SiO_2.3H_2O(s) + 3Ca(OH)_2(aq) \quad (4)$$

However, the reaction stoichiometry would not be exact as shown above. Usually gypsum ($CaSO_4$) is added during cement production. Gypsum avoids rapid setting of the cement. It reacts with tricalcium aluminate (C_3A) to form various aluminate and sulfoaluminate phases that are referred to as Ettringite phases. As examples:

$$3CaO.Al_2O_3(s) + 3CaSO_4(s) + 32H_2O(l) \rightarrow 3CaO.Al_2O_3.3CaSO_4.32H_2O(s) \quad (5)$$

$$3CaO.Al_2O_3(s) + 3CaSO_4(s) + 12H_2O(l) \rightarrow 3CaO.Al_2O_3.3CaSO_3.12H_2O(s) \quad (6)$$

'C_3A' and 'C_4AF' can also hydrate separately of calcium sulfate:

$$3CaO.Al_2O_3(s) + 6H_2O(l) \rightarrow 3CaO.Al_2O_3.6H_2O(s) \quad (7)$$

$$4CaO.Al_2O_3.Fe_2O_3(s) + 2Ca(OH)_2(aq) + 10\ H_2O(l) \rightarrow$$

$$3CaO.Al_2O_3.6H_2O(s) + 3CaO.Fe_2O_3.6H_2O(s) \quad (8)$$

Not long after the hydration starts, an induction period begins where the reaction slows drastically. This induction period is also referred to as the dormancy. Following the dormancy period, the reaction rate increases, where the nucleation and growth of the hydration products is dominant. At this stage, rapid hydration of 'C_3S' occurs, followed slowly by the hydration of 'C_2S':

$$2(2CaO.SiO_2)(s) + 4H_2O(aq) \rightarrow 3CaO.2SiO_2.3H_2O(s) + Ca(OH)_2(aq) \quad (9)$$

It is during this process that calcium hydroxide begins to precipitate as crystalline due to calcium hydroxide saturation; this phenomenon is referred to as Portlandite by cement chemists. The rate

of hydration slows and becomes diffusion controlled as the solution becomes concentrated with solid product. These reactions are slow but continue for weeks as the 'CSH' gel continues to form (MacLaren and White 2003). The heat produced by hydration can be detected, indicating that reactions continue slowly. The reactions could continue for years, as long as there is enough water and unreacted cement in the matrix. This continued reaction is desirable since strength and other desirable characteristics like low permeability can be achieved (Taylor et al. 2006).

Overall, hydration is the reaction of oxides in unhydrated cement in the presence of water. The following relationships of the stoichiometry is not precise, however, they are used to explain the hydration process of the oxides.

$$2(3CaO.Si0_2) + 6H_20 \rightarrow 3CaO.2Si0_2.3H_20 + 3Ca(OH)_2 \quad (10)$$

$$2(2CaO.Si0_2) + 4H_20 \rightarrow 3CaO.2Si0_2.3H_20 + Ca(OH)_2 \quad (11)$$

$$3CaO.\ A1_20_3 + 6H20 \rightarrow 3CaO\ A1_20_3\ 6H_20 \quad (12)$$

$$3CaO.Al_2O_3 + CaSO_4.2H_2O \rightarrow 3CaO.A1_2O_3.3CaSO_4.31H_2O \quad (13)$$

Calcium hydroxide provides most of the buffering capacity of cement due to its alkalinity. Insoluble calcium silicates deposit to form the cement matrix when calcium concentrations are greater than 100 mg/L and pH is above 11.0 (Shively et al. 1986).

2.4 POTENTIAL EFFECTS OF SURFACTANT ON HYDRATED CEMENT PROPERTIES

A detailed investigation of the effect of surfactants on hydrated cement products is not conducted. This section summarizes potential effects based on limited knowledge that the research team gained through literature review of surfactant properties and the extensive knowledge in cement chemistry. Hydration process continues in the presence of moisture, unhydrated cement, and favorable condition for reaction. Because of the hydrophilic structure, there is a possibility for surfactants to absorb and retain the moisture in the structure (Figure 6). The absorbed water may be released slowly and contribute to the long-term strength development. The long-term reaction leads to a stronger and less permeable product. On the other hand, if the absorbed water is released at a rate faster than optimum, it may lead to poor quality. These hypotheses need further investigations.

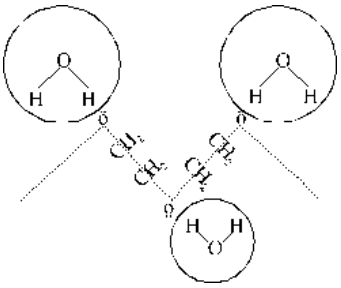


Figure 6. Schematic presentation of surfactants in water

2.5 POTENTIAL METHODS TO INVESTIGATE CEMENT, WPP, AND COMBINED STRUCTURE

As presented under research objectives, it is important to understand the chemical and physical properties of cement, WPP, and the final products that are made using cement-WPP mixture. Hence, experimental methods that are commonly used for such purposes are reviewed and listed in Table 2 (Hewlett 2004; Uchikawa 1993; Zofka et al. 2012).

Table 2. Potential Experimental Methods

Method
Xray powder diffraction
Optical microscopy
Scanning electron microscopy
Thermal analysis
XRF(X-Ray fluorescence)
Solution ion chromatography and atomic absorption (AA)
Emission spectroscopy
Gas chromatography (GC)
Liquid chromatography (LC)
Electron spectroscopy for chemical analysis (ESCA)
X-ray photoelectron spectroscopy (XPS)
ATR FT-IR Raman
Hg porosimetry
N_2 absorption technique (BET)

3 TESTING OF FRESH AND HARDENED MORTAR/GROUT PROPERTIES

3.1 INTRODUCTION

The testing for fresh and hardened properties of cement grout with waste powder paint (WPP) was performed by following the American Society for Testing and Materials (ASTM) standards. Table 5 shows the properties that were evaluated and their relevant ASTM standard(s). The SI units were utilized mostly to record and evaluate the test data following ASTM procedures.

Table 3. Mortar/Grout Properties and ASTM Standards

Property	ASTM Standard(s)
Heat of hydration	ASTM E 220 and ASTM C1074
Leaching	ASTM C 67
Shrinkage/Expansion (early age)	ASTM C 827
Absorption	ASTM C 67
Compressive strength (cube specimens)*	ASTM C192, ASTM C305, and ASTM C109
Drying shrinkage (beam specimens)*	ASTM C192, ASTM C305, and ASTM C596

* Cement mortar containing fine aggregate was used for evaluating the property

The Type-I Portland Cement was used as the cementitious material. Different percentages of the WPP were used as a cement replacement by weight. The 0% WPP mix was used as the reference.

The tests for (1) heat of hydration, (2) leaching, (3) shrinkage/expansion (early age), and (4) absorption were performed with mixtures without fine aggregate. These tests were conducted for the specimens at early hardening stage or with limited curing. Also, the compressive strength of some of these specimens was evaluated. The cube and beam specimens that were prepared for compressive strength test and drying shrinkage test, respectively, contained fine aggregate (sand). Compressive strength of cube specimens was conducted after curing them for 1, 3, 7, and 14 days. The drying shrinkage test of beam specimens was conducted for a duration ranging from 3 to 9 days.

3.2 HEAT OF HYDRATION

Heat of hydration is used to evaluate hydration rate to understand the rate of strength development. The details of the test parameters are shown in Table 6. Three equal size cylindrical containers were used for placing the grout. The containers were then filled with the respective mix (i.e., 0%, 10%, and 20% of WPP by weight). Even though the ASTM procedure is closely followed, the equipments used in this test are not the ones specified in the ASTM. The objective of performing this test was to develop an understanding of WPP contribution to the heat of hydration. Each mix was mixed for 3 min., weighed to 750 g and placed in the containers (Figure 7-a). Each of these cylindrical containers was placed inside another large insulated container (Figure 7-b) to create absolute adiabatic conditions for measuring the heat of hydration.

Table 4. Heat of Hydration Test Details

ASTM Standard	ASTM E 220, and ASTM C 1074		
W/C ratio	0.45		
Type-I cement (g)	600		
Water (ml)	270		
WPP as a cement replacement (%)	0	10	20
Mixing-water temperature (°C)	20.8	17.5	16.2
Weight of the test specimen (g)	750	750	750

(a) (b)

Figure 7. (a) Casting of specimens and (b) adiabatic container and data logger

Type K thermocouples were utilized along with a thermocouple input module (CB-7018) for this test. DASYLab® was used for logging the temperature from thermocouples at specified time intervals, and exporting to Microsoft-excel for analysis (Figure 8).

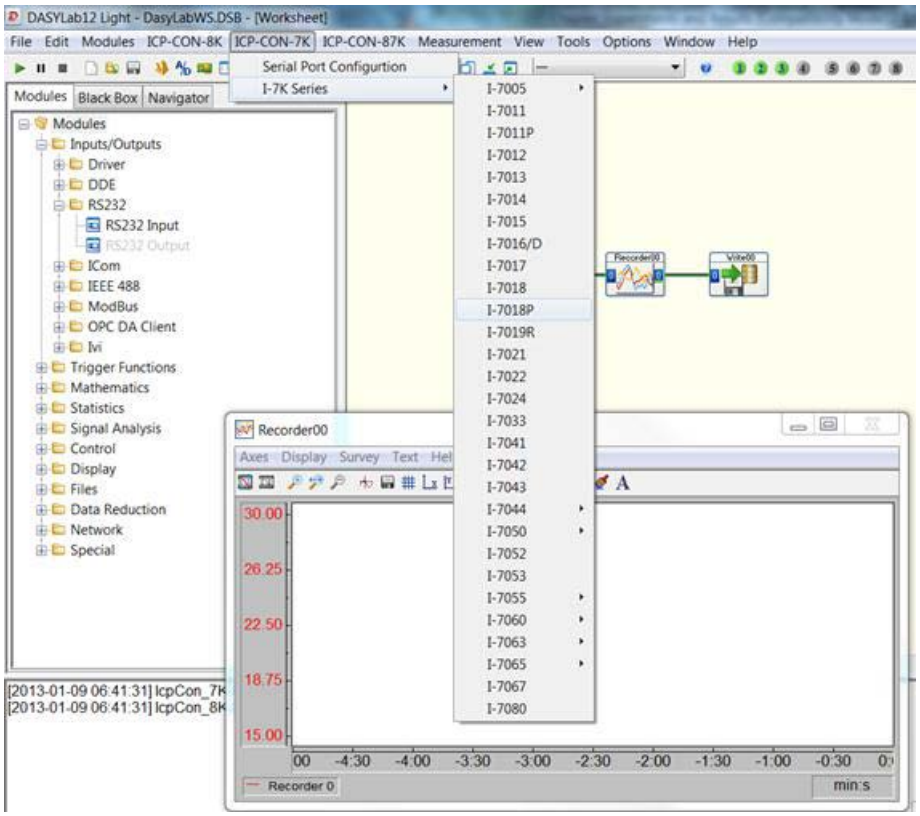


Figure 8. DASYLab® software interface

3.2.1 Calibration

Thermocouples were calibrated using the *comparison calibration* procedure from ASTM E 220. A water bath was used for calibration. The temperature of the water bath was raised to around 90°C and allowed to cool down gradually. In the first step of calibration, a digital thermometer was used to measure the temperature of a water bath at specified time intervals in parallel with the thermocouple. The average difference between the measured temperatures, from digital thermometer and the thermocouple was calculated and the *Cold Junction Temperature (CJC)* of the thermocouple input module was offset with the calculated amount.

In the second step of calibration, the calibrated thermocouple from the first step was used to calibrate the other thermocouples. The temperature of the water bath was again raised to around 90°C and allowed to cool down gradually. Temperature was measured with each of the thermocouples, including the calibrated thermocouple (i.e., Ch-0 in Figure 9). The Figure 9 shows the measured temperature of the water bath with respective thermocouples. The temperature difference for each thermocouple (i.e., Ch-1, Ch-2, and Ch-3 in Figure 9) with

respect to calibrated thermocouple (i.e., Ch-0) was calculated. The average temperature difference for thermocouples Ch-1, and Ch-2, was calculated as +0.2°C; whereas, for Ch-3 it was calculated as +0.0°C. These average values were added to respective thermocouple readings, thus, obtaining accurate temperatures.

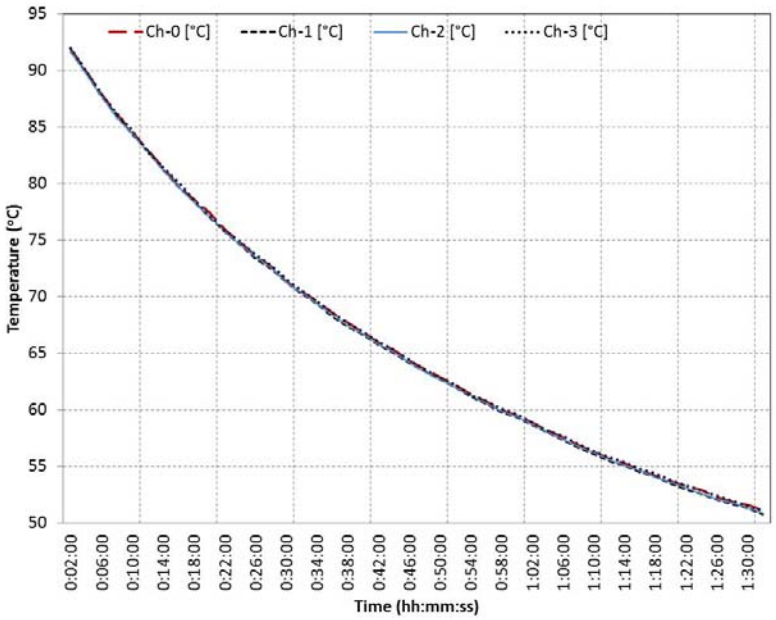


Figure 9. Calibration curve for four thermocouples

3.2.2 Testing

The thermocouple input module was connected with 4 thermocouples. Three of the thermocouples were embedded in the specimens containing 0%, 10%, and 20% WPP, and the fourth thermocouple was used to log the room temperature during the test. The thermocouples were embedded in the specimens at their mid depth. The test was started soon after embedding the thermocouples in the specimens.

The temperature data was collected every 1 min for a total duration of 7 days. The graph in Figure 10 shows corresponding hydration temperature of the specimens with 0%, 10%, 20% WPP, and room temperature with respect to time. The graph was plotted for about 65 hours only, because the temperature in the specimens remained almost constant after this time.

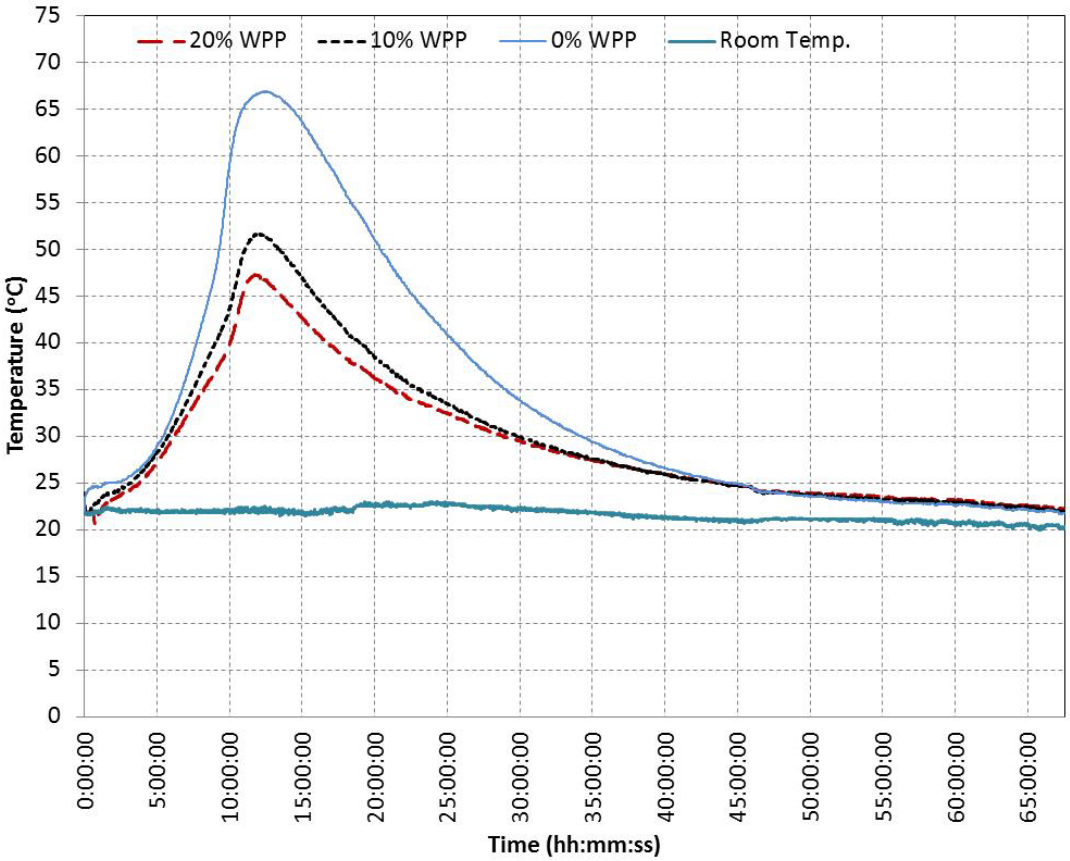


Figure 10. Heat of hydration variation in specimens with 0%, 10%, and 20% WPP

A typical heat of hydration curve is provided in the *Integrated Materials and Construction Practices for Concrete Pavement – Manual* (Taylor et al. 2006) (Figure 11). Comparing the test results depicted in Figure 10 with the typical curve shown in Figure 11, a *dormancy* period of around 2.5 hours along with nearly equal time for the *initial set* was observed for all the mixes. The *hydration* process of the 10% and 20% WPP mixes took approximately the same time as 0% WPP mix, but generated a less amount of head during hydration.

The temperature for the 0% reached a maximum of 67 °C; whereas, the 10% and 20% WPP specimens' temperature reached 52 °C and 47.5 °C, respectively. This shows a reduction of about 22.4% and 29.1% in the maximum hydration temperature for the 10% and 20% WPP mixes, respectively, compared to 0% WPP mix. But it was observed that the rate of decrease in temperature from the maximum peak, for 10% and 20% mixes, was relatively slower than the 0% mix. Therefore, all the mixes reached a constant temperature at approximately equal time. There was about 3 °C difference in mixing-water temperature of 10% and 20% mix with respect to 0% mix. This might have also contributed to the reduction in hydration temperature. However,

the difference observed with respect to the 0% mix is significant, but the difference between 10% and 20% is insignificant. In the *hardening* stage (Figure 11), the mix sets, begins to harden, and gains strength (Taylor et al. 2006). To identify the strength gained by the respective mixes, a compressive strength test was performed using the same specimens. The compressive strength test is discussed in the next section.

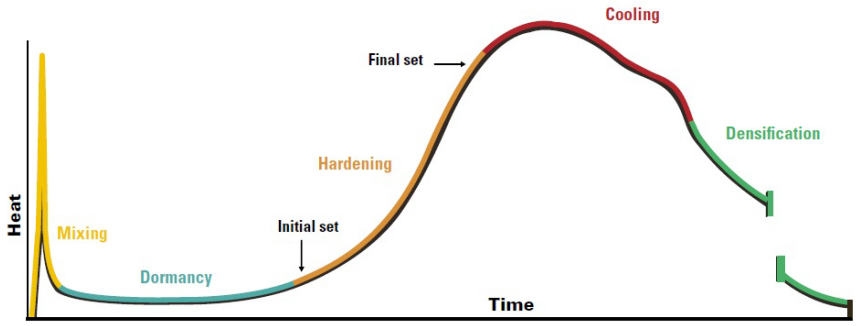


Figure 11. General hydration curve (Source: Taylor et al. 2006)

3.3 COMPRESSIVE STRENGTH – CYLINDRICAL SPECIMENS

The mixture of 0%, 10%, and 20% WPP was placed in cylindrical containers for the heat of hydration test. Therefore, the ASTM standard C 39 was used to test the compressive strength of those cylindrical specimens. The specimen parameters and the test data are presented in Table 7. The load was applied in displacement control mode and the rate of loading was of 0.05 cm/sec. The load as well as the deformation was recorded and the stress-strain curves for the 0%, 10%, and 20% WPP mix specimens were developed as shown in Figure 12.

Table 5. Details for Compressive Strength Test of Cylindrical Specimens

ASTM Standard	ASTM C 39		
W/C ratio	0.45		
Type-I cement (g)	600		
Water (ml)	270		
WPP as a cement replacement (%)	0	10	20
Mixing-water temperature (°C)	20.8	17.5	16.2
Weight of the test specimen (g)	749.5	717.5	715
Diameter of test specimen (avg.) (cm)	7.07	7.19	7.13
Height of test specimen (avg.) (cm)	7.86	8.07	8.09
Volume of test specimen (cm^3)	308.72	327.63	323.96

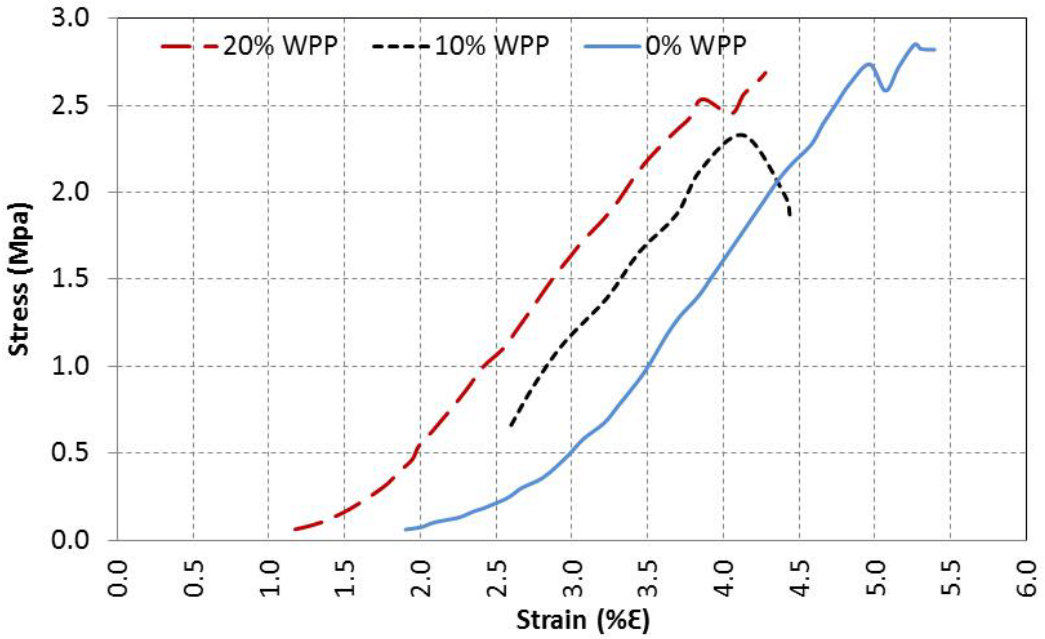


Figure 12. Stress-strain curves for the 0%, 10%, and 20% WPP mix specimens

The cylindrical specimens used for the heat of hydration test were kept in adiabatic conditions, without external curing, for up to 7 days. The compressive strength was performed on these specimens soon after the end of the 7 day period. The compressive strength for the 0%, 10%, and 20% WPP specimens was calculated as shown in Table 8. It was observed that the compressive strength values were very close for those specimens. This infers that the strength developed during the *hardening process* of the mixture, without external curing, is not affected by addition of WPP even though there was a reduction in the heat developed during hydration.

Table 6. Compressive Strength Calculation Results

Specimen	C/S area cm^2 (in^2)	Maximum Load kN (lbs)	Compressive strength MPa (psi)
0% WPP	39.27 (6.09)	10.74 (2415.05)	**2.74 (396.76)**
10% WPP	40.61 (6.30)	9.45 (2124.98)	**2.33 (337.57)**
20% WPP	40.00 (6.20)	10.15 (2280.79)	**2.54 (367.81)**

Further, investigating closely the texture of the crushed specimens (Figure 13-a), it was observed that the specimens with 10% and 20% WPP had developed an air-void system (Figure 13-c and d). This showed that there is more *air content* in the specimen compared to 0% WPP specimen (Figure 13-b). This infers that although the air content in 10% and 20% WPP specimens is large, they develop compressive strength near to the 0% WPP specimen that has very less air content.

The use of limited specimens in the tests, and the failure patterns suggest conducting further investigation to obtain quantitative results.

(a)

(b)

(c)

(d)

Figure 13. View of (a) specimens after compressive strength test, (b) 0% WPP mix specimen's texture, (c) 10% WPP mix specimen's texture and (d) 20% WPP mix specimen's texture

3.4 LEACHING TEST OF CYLINDRICAL SPECIMENS

The cylindrical specimens that were used for compressive strength test were used in the leaching test. The specimens were placed in porcelain bowls and were submerged up to equal height in de-ionized water. The ASTM standard C 67 was used for the test, but without a fan to expedite the evaporation. Initially the specimens were kept in a room with ambient temperature of 22 °C for up to 10 days, but, neither the leaching nor significant reduction in de-ionized water level was observed. Therefore, the specimens were transferred to the drying room with ambient temperature of 32.2 °C and the test was performed for additional 14 days. The details of the test are shown in Table 9.

Table 7. Details for Leaching Test of Cylindrical Specimens

Time interval	De-ionized water (ml)	Observation	Remarks
Day-1	400	Figure 14	Test started
Day-4	200		
Day-10	200		Drying temperature increased to 32.2°C
Day-11	250		
Day-13	500		
Day-16	250	Figure 15	
Day-20	500		
Day-24	0	Figure 16	Test ended

Images of the specimens were taken at start of the test (Figure 14), after 16 days (Figure 15), and at end of the test (Figure 16). After 16 days (i.e., after 6 days in drying room with 32.2 °C), efflorescence was observed on the cracked portions of 0% WPP specimen (Figure 15-a), but the 10% and 20% WPP specimens showed efflorescence at the bottom of the specimen (Figure 15-b and c). From this observation, it can be concluded that when water flows upward (capillary action) through the cracks in the 0% WPP specimen, the dissolved ions in the cement grout mixture are leached out making deposits on the concrete outer surface, showing the expected phenomenon of leaching. But, for the 10% and 20% WPP specimens, the dissolved ions in the cement grout mixture are discharged from the bottom surface. Because of the white color of the bowl, the efflorescence at bottom of the 10% and 20% WPP specimens is not clear in the images (Figure 15-b and c), but minute particles in water (Figure 15-b and c) signify presence of efflorescence. This phenomenon was confirmed by feeling the deposited particles near the bottom of specimens. Further, when the bottom surfaces of the specimens were investigated at the end of the test (i.e., after 14 days in drying room with 32.2^{o}C) (Figure 16), it was observed that 20% WPP specimen had more efflorescence than the 10% WPP specimen.

From the above observations, it can be anticipated that the seepage of water through capillary action is hindered due to addition of WPP. Thus, further investigation of WPP samples need to be performed for permeability and water repellency properties.

Figure 14. Specimens at start of leaching test

(a) (b) (c)

Figure 15. Specimens after 16 days (a) 0% WPP, (b) 10% WPP, and (c) 20% WPP

Figure 16. Specimens at the end of leaching test at 24th day

3.5 SHRINKAGE/EXPANSION – EARLY AGE

Generally, the cement grout is expected to shrink at the early age, which is called autogenous shrinkage. But during the casting of the cement grout specimens with WPP, an unusual phenomenon of cement grout expansion was observed (Figure 17). Therefore, a test was setup to measure the expansion of cement grout with 0%, 10%, and 20% WPP mixes. The test details are shown in Table 10. The test procedure was in accordance with ASTM standard C 827. However, adhering to the exact procedure as stipulated in the ASTM was impractical due to large volume expansion observed in the mixes with WPP (Figure 17-a). Hence, a special set up was developed incorporating digital dial gauges instead of the lasers.

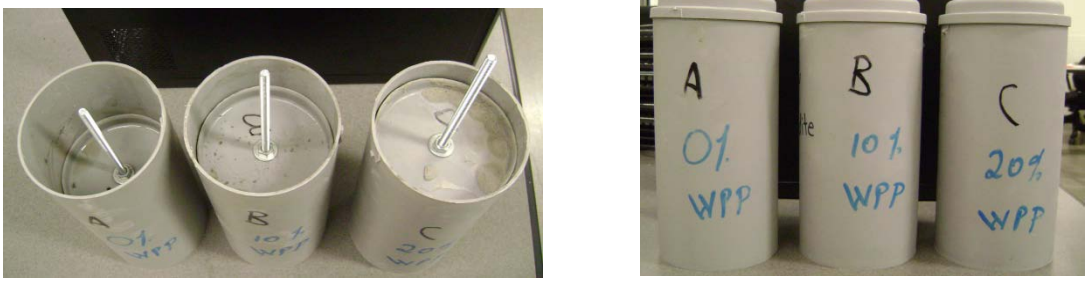
(a) (b)

Figure 17. View of unusual expansion of cement grout with WPP

Table 8. Details for Shrinkage/Expansion Test

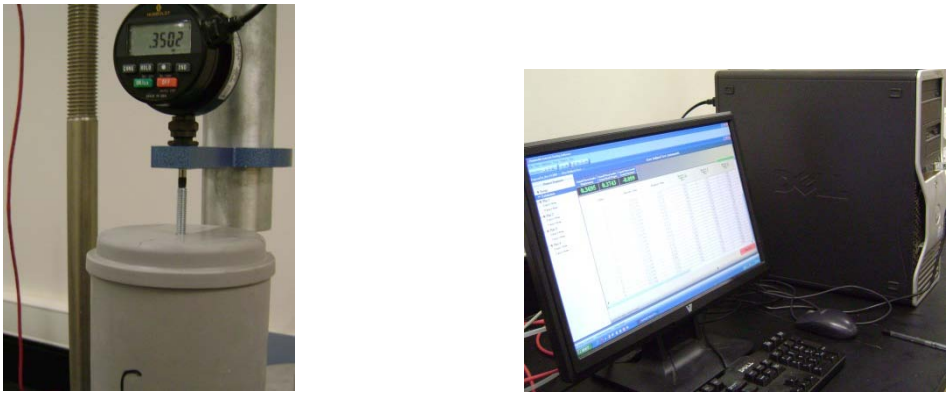
ASTM Standard	ASTM C 827		
W/C ratio	0.45		
Type-I cement (g)	1500		
Water (ml)	675		
Mixing duration (min)	3		
WPP as a cement replacement (%)	0	10	20
Mixing-water temperature (°C)	21.2	19.0	19.2
Mix temperature (°C)	24.3	22.8	22.2
Weight of the test specimen (g)	1700	1700	1700
Diameter of test specimen (cm)	10.16	10.16	10.16
Initial height of test specimen (cm)	11.40	12.30	12.70
Final height of test specimen (cm)	11.12	14.90	17.27

For the test, plastic lids acting as pistons were fabricated for three cylindrical containers (Figure 18-a). Another set of lids were fabricated to guide the vertical movement of the piston in each container (Figure 18-b). The digital dial gauges were mounted to measure the movement of the tip of piston (Figure 19-a). A data logging software by Humboldt® (Figure 19-b) was used to record the change in height of the specimen with respect to time.



(a) (b)

Figure 18. View of (a) cylindrical containers with plastic lids acting as pistons and (b) cylindrical containers with lids to guide the vertical movement of the piston



(a) (b)

Figure 19. View of (a) digital dial gauge for measuring the change in height of the specimen and (b) Humboldt® data logging software for recording data

Equal weight of cement grout mixture was used to cast 0%, 10%, and 20% WPP specimens (Table 10). The specimens with 0% and 10% WPP were prepared, digital dial gauges were mounted, and the data logger was started. The data was recorded for duration of 1 day. The change in height of the specimens was recorded for every 1.5 min and is plotted as shown in Figure 20. Only the initial 5 hour test data was plotted, because the shrinkage/expansion of the specimens was ceased and the data remained constant after this time.

The specimen with 0% WPP started shrinking soon after starting the test, whereas, the specimens with 10% and 20% WPP started expanding after a delay of about 10 to 15 min after starting the test (Figure 20). Further, the expansion of the specimens with 10% and 20% WPP ceased after 2.5 hours. Moreover, considering the data obtained from heat of hydration curve (Figure 10), the initial setting occurred after the dormancy period of 2.5 hours. Thus, it can be inferred that the expansion occurred before the initial setting.

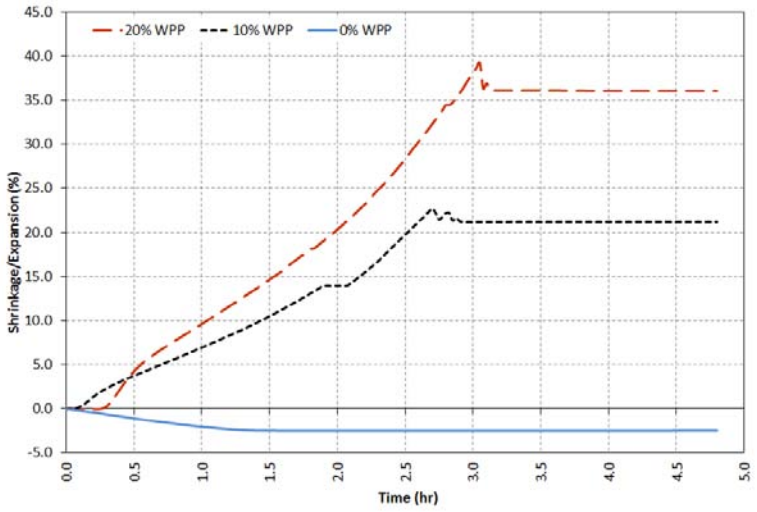


Figure 20. Percentage change in height of 0%, 10%, and 20% WPP mix specimens

The 0% WPP specimen shrank about 2.45% during the initial 2 hour period, and then its height remained constant until the end of the test (Figure 20). The specimen with 10% WPP expanded about 22.76% during the initial 2.75 hours, shrank about 1.63% in next 15 min, and maintained a constant height until the end of the test (Figure 20). Similarly, the specimen with 20% WPP expanded about 39.21% during the initial 3 hour period shrank about 3.23% in next 10 min, and maintained a constant height until the end of the test (Figure 20).

The effective expansion in 0% WPP specimen was -2.45%, whereas, the effective expansion in 10% and 20% WPP specimens was 21.13% and 35.98%, respectively (Table 11). The significant change in the height of the specimens can be noticed by observing the elevation of their respective piston tip in Figure 21.

Thus, from this test, the innovative property of WPP to expand the cement grout was identified. This WPP property opposes the shrinkage property of regular cement grout. This prompts to initiate a full scale research to identify *several potential applications* of the expanding property of WPP in cement grout mixture.

Table 9. Summary of the Early Age Shrinkage/Expansion Test

Weight % of WPP	Effective expansion (%)
0%	-2.45
10%	21.13
20%	35.98

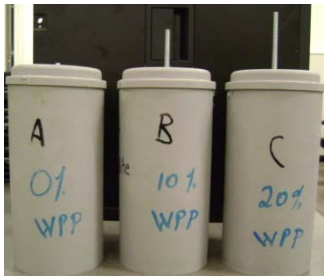


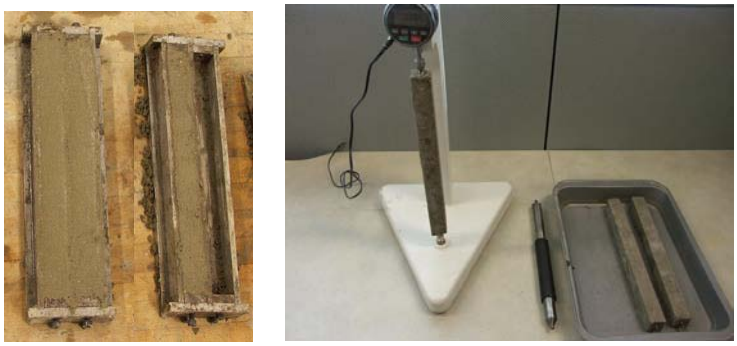
Figure 21. Piston tip elevation of 0%, 10% and 20% WPP mix after 1 day

3.6 DRYING SHRINKAGE OF MORTAR

The drying shrinkage test was performed in accordance with the ASTM standards and the mix proportions are listed in the Table 12. Three sets of specimens are prepared with 0%, 5%, and 10% of WPP as cement replacement by weight. Each set contained 3 specimens. The specimens and the apparatus are shown in Figure 22. Measurements were taken at 3, 4, 5, 6, 7, and 9 days after curing for only 24 hours.

Table 10. Details for Drying Shrinkage Test of Cuboid Specimens

ASTM Standard	ASTM C192, ASTM C305, and ASTM C596		
W/C ratio	0.45		
Mix proportion (cement **:** sand)	1 **:** 2.75		
Type-I cement (g)	740		
Sand (passing # 4 sieve) (g)	2035		
Water (ml)	333		
WPP as a cement replacement (%)	0	5	10



(a) (b)

Figure 22. a) Shrinkage test specimen preparation, b) Shrinkage testing instrument

The drying shrinkage results from 3 specimens of each sample set were averaged and the percentage drying shrinkage was calculated as shown in Table 13. The change in average drying shrinkage of 0%, 5% and 10% WPP specimens is shown in Figure 23.

Table 11. Drying Shrinkage Test Results

Age (days)	WPP (%)	Sample no.	Drying shrinkage (in.)	Average drying shrinkage (in.)	Drying Shrinkage (%)
3	0	1	0.21	0.23	2.26%
3	0	2	0.24		
3	0	3	0.23		
3	5	1	0.18	0.20	1.97%
3	5	2	0.20		
3	5	3	0.21		
3	10	1	0.23	0.20	1.98%
3	10	2	0.19		
3	10	3	0.18		
4	0	1	0.22	0.22	2.23%
4	0	2	0.23		
4	0	3	0.21		
4	5	1	0.18	0.19	1.94%
4	5	2	0.20		
4	5	3	0.21		
4	10	1	0.23	0.20	1.97%
4	10	2	0.19		
4	10	3	0.17		
5	0	1	0.21	0.22	2.20%
5	0	2	0.23		
5	0	3	0.22		
5	5	1	0.18	0.19	1.93%
5	5	2	0.19		
5	5	3	0.21		
5	10	1	0.23	0.20	1.97%
5	10	2	0.19		
5	10	3	0.17		
6	0	1	0.20	0.22	2.20%
6	0	2	0.23		
6	0	3	0.23		
6	5	1	0.18	0.19	1.93%

6	5	2	0.19		
6	5	3	0.21		
6	10	1	0.22	0.20	2.05%
6	10	2	0.19		
6	10	3	-		
7	0	1	0.21	0.22	2.19%
7	0	2	0.23		
7	0	3	0.22		
7	5	1	0.18	0.20	1.97%
7	5	2	0.20		
7	5	3	0.21		
7	10	1	0.22	0.20	2.03%
7	10	2	0.18		
7	10	3	-		
9	0	1	0.20	0.22	2.18%
9	0	2	0.23		
9	0	3	0.22		
9	5	1	0.17	0.19	1.92%
9	5	2	0.19		
9	5	3	0.21		
9	10	1	0.22	0.20	2.03%
9	10	2	0.18		
9	10	3	-		

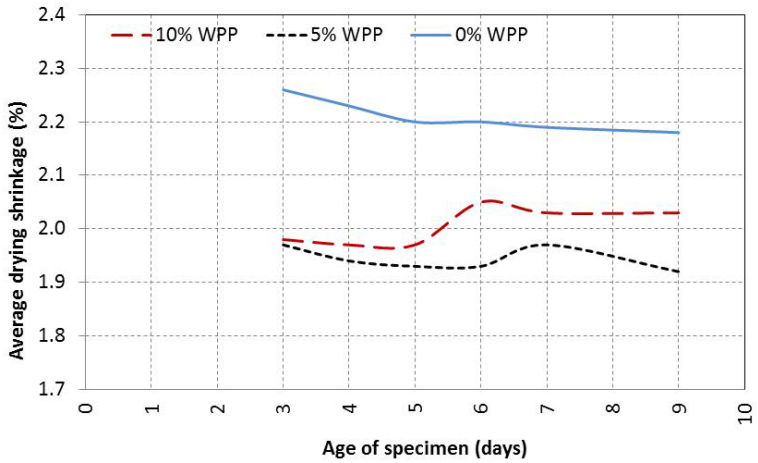


Figure 23. Average drying shrinkage of 0%, 5% and 10% WPP specimens

The average drying shrinkage graph (Figure 23) of 0% WPP shows decrease in drying shrinkage percentage over the experiment duration. And the graphs for mixtures with 5% and 10% WPP (Figure 23) show decrease in drying shrinkage percentage for up to 5-6 days and then an increase in drying shrinkage percentage at around 6-7 days. However, the graph does not show any particular trend and hence, data for further durations need to be acquired, so that, there will be sufficient data points to make inferences.

3.7 ABSORPTION – CYLINDRICAL SPECIMENS

The specimens with 0%, 10%, and 20% WPP by weight were prepared in a room with ambient temperature of 22 °C (Figure 24). The absorption test of the cylindrical specimens was performed in accordance with ASTM standard C 67 and the details are shown in Table 14. The specimens shown in Figure 24 were cut into two portions and used for the absorption test (Figure 25-a). The labels and dimensions of the specimens used for the absorption test are shown in Table 15. The specimens were prepared allowing one-dimensional free shrinkage/expansion. Each cylindrical specimen was cut into two and the top portion was labeled with an asterisk (Table 15).

Figure 24. Specimens with 0%, 10%, and 20% WPP mix

Table 12. Details for Absorption Test of Cylindrical Specimens

ASTM Standard	ASTM C 67		
W/C ratio	0.45		
Type-I cement (g)	1250		
Water (ml)	562.5		
Mixing duration (min)	3		
WPP as a cement replacement (%)	0	10	20
Weight of the test specimen (g)	1100	1100	1100

Table 13. Labels and Dimensions of the Specimens used in Absorption Test

Specimen	Height (cm)	Diameter (cm)	Volume (cm^3)
0% - A	5.32	7.64	243.51
0% - B*	5.62	7.65	257.93
10% - A*	6.51	7.70	303.32
10% - B	6.30	7.59	285.13
20% - A*	6.65	7.67	307.42
20% - B	6.88	7.62	313.65

* represents the top portion of the respective cylindrical specimen

(a)

(b)

(c)

Figure 25. View of specimens (a) after preparation for absorption test (b) during the drying process and (c) submerged in water

The WPP is a polymer and the effect of drying temperature on WPP in grout is not known. Hence, the test was performed in three trials (viz., trial–1, trial–2, and trial–3) to identify the effect of drying temperature on the absorption of the specimens. In trial–1, the drying temperature was set to 65.5 °C and the specimens were dried for 24 hours (Figure 25-b). Then the specimens were allowed to cool down to room temperature and weighed to obtain the dry weight. Afterwards, the specimens were submerged in water with temperature of around 22.5 °C, for 24 hours for absorption (Figure 25-c). The specimens were removed from the water and prepared to saturated surface dry (SSD) condition. The absorption is calculated for the trial–1 as shown in Table 16.

Table 14. Absorption Test Trial–1 Results

Specimen	Weight (g)		Absorption (%)
	Dry	SSD	
0% - A	416.5	461.0	**10.68**
0% - B*	431.5	487.0	**12.86**
10% - A*	357.0	440.0	**23.25**
10% - B	392.5	451.0	**14.90**
20% - A*	338.0	424.0	**25.44**
20% - B	396.5	462.0	**16.52**

In trial–2, the drying temperature was set again to 65.5 °C and the specimens were dried for 24 hours. After weighing the dry weight, the specimens were submerged in water with temperature of around 20.3 °C. After documenting the SSD weight, the absorption was calculated as shown in Table 17. The trial–1 and trial–2 were performed to ensure accurate identification of the absorption by averaging the two trials as shown in Table 18. These two trials were performed with nominal drying temperature of 65.5 °C at which the WPP will be in its *rubber-elastic state* (Graewe and Rettig 2002). To investigate the change in absorption due to increase in drying temperature, trial–3 was performed with maximum limit of the drying temperature specified in ASTM standard, and is discussed in later text.

Table 15. Absorption Test Trial–2 Results

Specimen	Weight (g)		
	Dry	SSD	Absorption (%)
0% - A	412.0	461.5	**12.00**
0% - B*	429.5	487.5	**13.50**
10% - A*	357.0	439.5	**23.11**
10% - B	390.0	451.5	**15.77**
20% - A*	338.5	422.5	**24.81**
20% - B	395.0	462.5	**17.09**

Table 16. Average Absorption from Trial–1 and Trial–2

Specimen	Absorption (%)		
	Trial–1	Trial–2	Average
0% - A	10.68	12.00	**11.34**
0% - B*	12.86	13.50	**13.18**
10% - A*	23.25	23.11	**23.18**
10% - B	14.90	15.77	**15.34**
20% - A*	25.44	24.81	**25.13**
20% - B	16.52	17.09	**16.81**

* represents the top portion of the respective cylindrical specimen

The average absorption result from trial–1 and trial–2 shows that the absorption in the specimen increases with increase in the percentage of WPP content in the mix (Table 18). Further, the top portion of the original specimens (i.e., from shrinkage/expansion test) shows greater absorption than the bottom portion. Thus, it can be inferred that the amount of constraint provided for free expansion may alter the microstructure. There is a possibility to reduce permeability by providing adequate restrain to the expansion of the grout with WPP. However, the possibility of developing large stresses on the formwork due to expansion property of WPP need to be considered and calls for further investigation.

For the trial–3, the drying temperature was set to 115 °C and the specimens were dried for 24 hours. After measuring the dry weight, the specimens were submerged in water with temperature of around 22 °C for 24 hours. After documenting the SSD weight, the absorption was calculated (Table 19). As expected, there was a change in absorption characteristics compared to trial–1 and trial–2.

Table 17. Absorption Test Trial–3 Results

Specimen	Weight (g)		Absorption (%)
	Dry	SSD	
0% - A	369.5	461.0	**24.76**
0% - B*	385.0	487.0	**26.49**
10% - A*	330.5	437.5	**32.37**
10% - B	353.5	445.5	**26.02**
20% - A*	315.5	413.0	**30.90**
20% - B	358.5	446.0	**24.41**

The 115 °C drying temperature for this trial–3 was within the *melting state* temperature range of WPP (Graewe and Rettig 2002). To identify if there is any effect of change in WPP state on absorption, the average increase in absorption with respect to trial–1 and trial–2 was calculated (Table 20). The results show a decrease in extra absorption with increase in WPP content. From this result it can be inferred that the increase in drying temperature changes the WPP state possibly to a *melting state* that affects the absorption properties of cement grout with WPP. Thus, presence of WPP is possibly resulting in a reduction in the absorption when dried/cured at high temperatures. But this phenomenon need to be further investigated with several trials of increased drying temperature, possibly up to 190°C (i.e., beyond the ASTM standard drying temperature limits) to capture the effect of *WPP in melting state* on the absorption of the specimen.

Table 18. Calculation for Extra Absorption in Trial–3 with respect to Trial–1 and Trial–2

Specimen	Extra Absorption (%)		
	Trial 3 – Trial 1	Trial 3 – Trial 2	Average
0% - A	14.08	12.76	**13.42**
0% - B*	13.63	12.99	**13.31**
10% - A*	9.12	9.26	**9.19**
10% - B	11.12	10.25	**10.69**
20% - A*	5.46	6.09	**5.78**
20% - B	7.89	7.32	**7.61**

3.8 COMPRESSIVE STRENGTH – CUBE SPECIMENS

Compressive strength testing was performed according to ASTM standards and the testing parameters are provided below in Table 21. Three sets of samples were prepared with 0%, 5%, and 10% of WPP as cement replacement by weight. Each set contained 3 specimens. The casting mold and the specimens are shown in Figure 26. The specimens were tested after 1, 3, 7, and 14 days of curing and the average compressive of the specimens were calculated as per the ASTM (Table 22).

Table 19. Details for Compressive Strength Test of Cube Specimens

ASTM Standard	ASTM C192, ASTM C305, and ASTM C109		
W/C ratio	0.45		
Mix proportion (cement **:** sand)	1 **:** 2.75		
Type-I cement (g)	740		
Sand (passing # 4 sieve) (g)	2035		
Water (ml)	333		
WPP as a cement replacement (%)	0	5	10

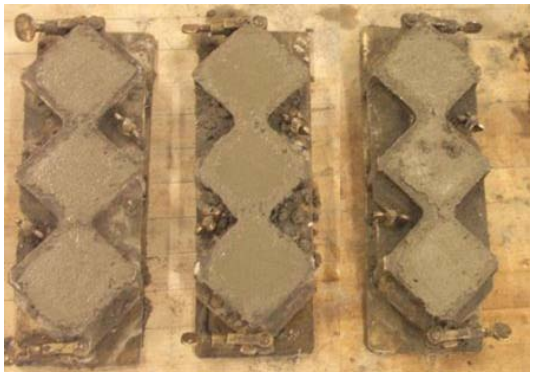


Figure 26. Compressive strength test specimen preparation

Table 20. Compressive Strength Test Results

Age (days)	WPP (%)	Sample no.	Compressive strength (MPa)	Avg. compressive strength (MPa)
1	0	1	-	9.4
1	0	2	10.0	
1	0	3	8.8	
1	5	1	9.8	9.4
1	5	2	10.0	
1	5	3	8.3	
1	10	1	-	10.0
1	10	2	7.3	
1	10	3	12.7	
3	0	1	23.5	23.2
3	0	2	-	

3	0	3	22.9	
3	5	1	13.8	13.1
3	5	2	12.0	
3	5	3	13.5	
3	10	1	-	-
3	10	2	-	
3	10	3	-	
7	0	1	37.4	34.7
7	0	2	32.8	
7	0	3	33.9	
7	5	1	25.2	27.8
7	5	2	29.5	
7	5	3	28.7	
7	10	1	13.4	14.6
7	10	2	16.0	
7	10	3	14.5	
14	0	1	41.2	37.3
14	0	2	32.4	
14	0	3	38.4	
14	5	1	28.7	28.1
14	5	2	25.7	
14	5	3	30.0	
14	10	1	19.4	19.2
14	10	2	19.0	
14	10	3	19.1	

(-) indicates that the specimens were faulty and their results were discarded

A graphical representation of the average compressive strength test results of 0%, 5%, and 10% WPP is shown in Figure 27. It can be observed that the compressive strength values of specimens with 0% and 5% WPP reaches a constant value after 7 days of curing. Whereas, the compressive strength of specimens with 10% WPP did not reach a constant value; thus, further investigation of curing practices is needed. The results show a decrease in compressive strength with increase in WPP content. The potential reasons for the reduction in compressive strength can be the following:

- The WPP was added as a cement replacement, thus, *the amount of cement is reduced in the specimens as it is replaced with 5% and 10% WPP*. Hence, lower compressive strength values infer that the WPP did not contribute to compressive strength.

- After the compressive strength test of cylindrical specimens (Section 3.3), it was observed that the specimens with WPP consisted of significant amount of air-voids compared to specimen without WPP. Further, the early age shrinkage/expansion test results (Section 3.6) indicated that the specimens with WPP expand significantly at early age itself. Thus, from the aforementioned observations it can be concluded that when the cube specimens were prepared and leveled at top to obtain a standard volume of 131 cm^3 (8 in^3), with respect to standards, *the specimens with WPP attained less density of mixture compared to specimens without WPP (i.e., equal volumes but unequal masses)*.

- *The WPP is a polymeric substance that may have affected the crystalline formation of the cement matrix*, thus reducing the compressive strength of the specimens. Surfactants in WPP may alter water absorption and retention characteristics within the mix leading to change in compressive strength.

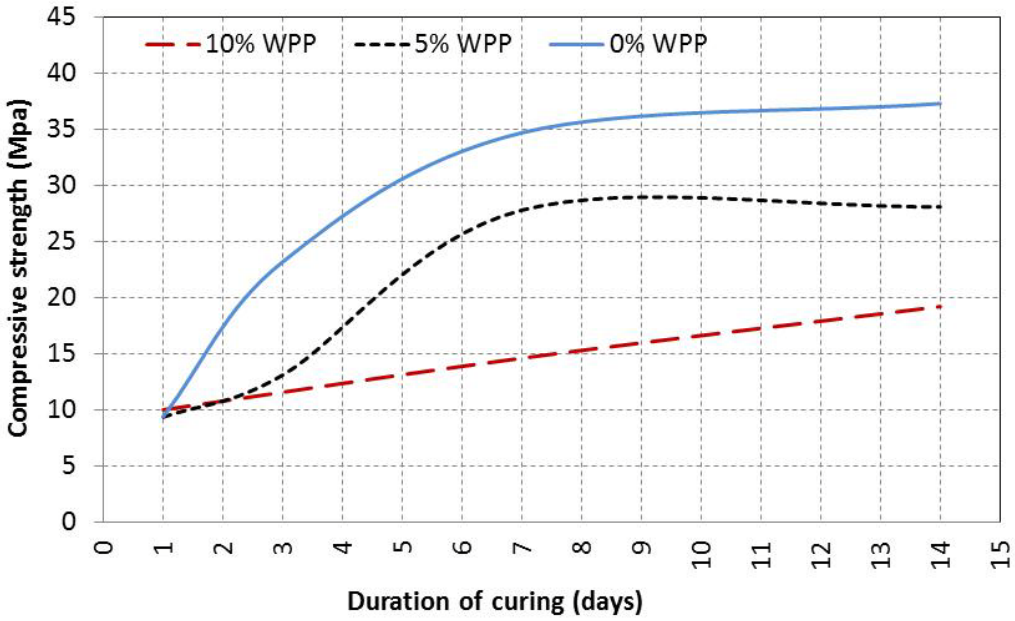


Figure 27. Compressive strength curves after 14 days of curing for specimens with 0%, 5%, and 10% WPP

4 SUMMARY AND CONCLUSION

The cement-based products use a wide range of polymeric admixtures to improve the fresh and hardened properties of concrete. The WPP that is basically made of different types of polymers is available in abundant quantity from auto and furniture industries' discharge. This material is usually dumped into the landfills. To help sustainability, it is vital to explore potential use of WPP in other industries.

The use of waste powder paint (WPP) in cement-based products such as grout and mortar was evaluated by analyzing their fresh and hardened properties. This project was limited to the testing of WPP as a cement replacement. It is demonstrated from the limited investigations conducted during this project that WPP can be effectively used in cement-based products where expansive and water repellency properties are required.

The summary of results from the fresh and hardened property tests is provided below.

1. **Heat of hydration:** This test was performed on grout mix specimens with and without WPP as cement replacement for the duration starting from fresh state of the specimens lasting up to 7-days of hardened state. The specimens with and without WPP showed almost equal time for the initial set. However, it was observed that the maximum temperature peak during the hydration process of a specimen is reduced with increase in the WPP content. Also, it was observed that the rate of decrease in temperature from the maximum temperature peak, for specimens with WPP, was relatively slower than the specimen without WPP. This eventually constituted to approximately equal durations for cement hydration process in those two types of specimens. This result encouraged to test the compressive strength to identify the effect of the observed temperature profiles on their respective strength development.
2. **Compressive strength:** This test was performed for the cylindrical specimens obtained from the heat of hydration test as well as for the standard 2 in. cube specimens. The cylindrical specimens were tested soon after the end of heat of hydration test (i.e. after 7 days). These specimens were kept in adiabatic conditions, without external curing. The test results for these specimens showed very close compressive strength values. Thus, it can be concluded that the strength developed during the hardening process of a specimen, without external curing, is not affected by addition of WPP even though the heat of

hydration peak drops. It should be noted that the specimens in this test had almost equal amount of mass but unequal volumes.

The compressive strength test of cube specimens with 0% and 5% WPP by weight revealed that with increase in curing duration the strength increases and becomes constant after a certain period of time. The specimens without WPP and with comparable curing durations achieved the greatest compressive strength compared to the specimens with WPP. Specimens with 5% WPP exhibited a similar trend in strength development, but with lower strength values compared to the specimens with 0% WPP. However, specimens with 10% WPP as cement replacement showed continuous rate of strength development during the entire duration of 14 day moist curing. However, the strength with WPP is always less than the strength of specimens without WPP.

3. **Leaching test:** The leaching test was performed using grout specimens. After performing the leaching test for 16 days, it was observed that the dissolved ions in the cement grout leached out making deposits on sides of the specimen without WPP. After 24 days, the specimens with WPP showed efflorescence at the bottom surface. Further, the efflorescence at the bottom surface of the specimen was greater with increased content of WPP. These observations left several questions unanswered. (1) What material in WPP does cause leaching? (2) What is the leaching mechanism of specimens with WPP? However, the test results show a potential of using WPP to minimize capillary suction.
4. **Early age shrinkage/expansion test:** During the casting of cement grout specimens with WPP, an unusual phenomenon of cement grout expansion was observed. Thus, this special test for early age shrinkage/expansion evaluation was initiated using dial gauges. The experiment was conducted for duration of 1-day. The specimen without WPP started shrinking soon after starting the test, whereas, the specimens with WPP started expanding after a delay of about 10 to 15 min after starting the test. The shrinkage of specimen without WPP ceased after 2 hours, whereas the expansion of the specimens with WPP ceased after 2.5 to 3 hours. The specimens with WPP had a minute shrinkage following the large expansion. From the test results it was inferred that the expansion occurred before the initial setting. There is a great potential to use WPP to develop expansive grout to mitigate many issues that are observed due to shrinkage.

5. **Drying shrinkage test:** The drying shrinkage test was performed following 24 hours of curing. A continuous decrease in drying shrinkage percentage was observed for the specimen without WPP with increase in its age. Whereas, for the specimens with WPP, the drying shrinkage percentage decreased up to 5-6 days and then an increase in the drying shrinkage percentage was observed at around 6-7 days. However, the results did not demonstrate any specific trend; thus requires additional drying shrinkage data before making any inferences.
6. **Absorption test:** As inferred from pervious tests result, the cement grout specimen with WPP exhibits lower density due to expansion than the specimen without WPP. Hence, it provides more porous cement matrix. The absorption test conducted with nominal drying temperature of 65.5°C (i.e., in rubber-elastic state temperature range of WPP) revealed that the absorption percentage increases with increase in WPP content in a specimen. Further, another set of absorption test was performed with drying temperature of 115°C (i.e., in melting state temperature range of WPP). This set of test revealed an unexpected phenomenon of decrease in absorption with increase in WPP content, thus it was concluded that drying/curing a cement grout specimen with WPP at temperature that is in the melting state temperature range of WPP, is eventually reducing the absorption properties of the specimen.

   The porosity and water repellency evaluation with absorption test shows that the porosity of the cement grout with WPP can be improved by heating up the sample to a temperature that is in the melting state temperature range of WPP (i.e., 90°C to 190°C). However, the microstructure of specimen needs to be analyzed with optical or electron microscopy to identify any other effects of WPP phase transition.

5 FUTURE WORK

This project can be further extended to find out potential applications in civil engineering construction. Hence, the following suggestions are made as future research work.

- The WPP was added in powder/solid form for all the experiments conducted in this research. The liquid WPP form is not experimented due to the lack of knowledge in the appropriate solvent for WPP to dissolve. And also the actual chemical reactions between the cement and WPP in liquid form need to be understood from future research work.

- X-Ray diffraction can be used to identify the composition of the WPP and the final product. Various samples/specimens with and without WPP in different percentages, before and after expansion can be analyzed.
- SEM images can be used to experiment the microstructure of the WPP, cement, and the final cement-based product with and without WPP. The samples/specimens at different depths before and after expansion can be investigated to aid the experimental results.
- Samples with different WPP replacement percentages can be used to check the shrinkage or expansion. Also, the impact of adding aggregates to the cement mixture with different mix proportions can be tested.
- The bending behavior of the samples with 3-point loaded beams (i.e., check for flexural strength of the beams due to the addition of the WPP). The use of fibers in cement-based products has been experimented and widely used in civil engineering industry. This phenomenon can be correlated with the use of WPP which is basically a polymer that is not water soluble. From the test results of using dry powder/solid form WPP with cement has shown that there is no active reactions in the final product. Hence, this mixture can be tested for bending or flexibility behavior and tensile strength of the cement-product. Experiments for bending behavior of the sample beams and tensile strength tests can be also be performed for different WPP mix proportions.
- Even though ASTM test methods were adopted for this research project, alternate procedures or modifications to the existing standards are required to evaluate the specific characteristics of the mix.
- Comprehensive mixes need to be developed for field applications.

The outcome of the research could allow WPP to be used in the following civil engineering applications.

1. Grouting of connection between precast elements when there is a need of non-shrink/expansive filling material.
2. Filling voids with restricted access by pumping or with gravity flow.
3. Underpinning of old structure foundations, when there is a need for less dense cementitious material with expansive nature. The compressive strength is not a critical parameter for such applications.

4. Filling masonry wall cavities with expansive grout or mortar.
5. Making masonry blocks and paving/cladding stones. However, this requires further investigation into the WPP chemicals that affect water repellency, absorption, and leaching due to their continuous exposure to moisture..

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