

The Influence of Commonly Used Plasticizing Admixtures on the Plasticizing Effect of Cement Paste

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Abstract – The article aims to present a research into the impact of dosage and effectiveness of commonly used plasticizing admixtures. More specifically, it focuses on rheological properties of the cement pastes (yield stresses and plastic viscosities) of different testing times (from 0 min to 90 min after mixing). The following materials were used in the study: Portland cement CEM I 42.5 R, plasticizer P (ligno-sulphonates based), superplasticizer SP1 (modified acrylic polymer based), superplasticizer SP2 (polycarboxylate esters based) and water. Experiments were carried out using rotational rheometer Rheotest RN4.1 with coaxial cylinders. It was observed that larger dosage of SP1 (0.6–1.2 %) results in slower increase in plasticizing effects until the 90 min margin. In conclusion, from the start of mixing until the 90 min margin, the best plasticizing effect and its retention are achieved by superplasticizer SP2. Recommended SP2 dosage varies within the range of 0.6 % to 0.8 %.

Keywords – Portland cement paste, yield stress, plastic viscosity, plasticizing admixtures, rheological properties.

I. INTRODUCTION

These days, concretes such as self-compacting concrete (SCC), ultra-high performance concrete (UHPC), and eco-friendly concretes have been extensively developed and their use is increasing. Generally, the quality of the particular concrete depends on the quality of each constituent ingredient used in the mixture. In order to improve the quality and optimize the properties of these concretes (i.e. ease of placement), the control of workability becomes crucial [1], [2].

Industrial and structural requirements are subject to immense discrepancies and variations, and the fresh concrete mixture must thus be adequately prepared for many operational stages and processes including mixing, transportation, handling, pumping, casting, consolidation and finishing. Since the cost of those processes, especially on the industrial scale, is significant, it is immensely beneficial to increase workability and prolong the period of time when mistakes can still be rectified relatively easily. Thus, plasticizing admixtures have become key ingredients of prepared modern concretes.

The main purpose of using plasticizing admixtures is to increase the fluidity without adding excessive amounts of water. As Gelardi and Flatt [3] explained, at the lowest, molecular, level, plasticizing admixtures “physically separate the cement particles by opposing their attractive forces with steric and/or electrostatic forces”. That, consequently, makes the fresh concrete mixture easier to work, place and shape.

The most commonly used plasticizing admixtures are ligno-sulphonates (P), synthetic polymers, such as modified acrylic polymers (SP1) and polycarboxylates (SP2) [4]. Ps, which have a limited water reduction ability of ~ 10 %, are mainly used to enhance the fluidity retention in ready-mix applications. By contrast, synthetic polymers (SP1, SP2) have exceptionally versatile chemical structures and can achieve water reduction of up to 40 %.

A number of researchers have indicated that fresh concrete can gradually lose its workability [5]. The extent, to which this effect manifests, depends on the type and dosage of the admixture and the mixing procedure. Workability can be maintained for a long period of time (1–2 hours or more) [6]. This is commonly attributed to two potential causes: the reserve of polymer in solution can interact with new created surfaces over time and preferential interaction of polymers with aluminates hydrating faster than silicates in the first hours of hydration [6]. In both cases, high surface coverages – and therefore workability – are maintained longer.

For a long time, the general terms used to characterize the property of a concrete to be placed with ease were consistency, workability or flowability. These general terms have been used to reflect personal viewpoints rather than scientific precision [1], [2], [7]–[9]. The slump test (according to EN 12350-2) is a commonly used method to evaluate the workability of the fresh concrete. It is a very simple test that simulates adequately the flow behaviour of concrete in a quasi-static regime that directly relates to yield stress [10]. Notably, several studies have shown that slump value is a necessary but not a sufficient measurement to characterize the fundamental rheological properties of concrete [11]–[13].

From the empirical measuring standpoint, concrete is a multiscale material in which cement paste may be considered to be a dense suspension of cement particles in water (i.e.: flocculated system due to Van der Waals attraction and hydrodynamic impacts) [14]. Cement pastes are attributed to Bingham systems, the flow of which is described by two rheological parameters – plastic viscosity μ_{pl} [15]–[17] and yield stress τ_0 (below which the suspension of cement particles in water displays solid-like behaviour).

Plastic viscosity is related to the number and size of flocculated particles, while the yield stress is a measure of strength and number of inter-flocculated particle interrelations that are broken down when shear is applied [18], [19]. The microstructure of flocculated cement particles to which high yield stress is

attributable is the three-dimensional network that often forms through flocculation. The yield stress thus reflects the extent of this flocculation and the strength of the attractive interparticle forces responsible for the flocculation [20].

A shear thinning behavior in plasticized systems is generally reported in a rich body of the literature [21]–[24]. Such behaviour results from the above-mentioned yield stress. A linear dependence of shear stress τ on shear rate $\dot{\gamma}$ is often observed, which makes the Bingham model the most common one for this purpose. The slope of this linear regime is referred to as the “plastic viscosity μ_{pl} .” [25].

In relation to plastic viscosity, Hot et al. [26] found that viscous dissipation decreases with increasing polymer adsorption. This implies that the dependence of plastic viscosity is inherently affected by plasticizing admixtures, notably much less than yield stress. This occurs largely because of the ability of plasticizing admixtures to keep particles separated. An additional effect – the nonadsorbed plasticizing admixture residue – is hard to mitigate but has less practical importance.

A number of methodologies can be used to determine rheological properties of cement paste. For this purpose, various rheometers could be used, which vary according to the mode of operation [27]–[31]. One of the most widespread cement paste rheological characterization techniques is based on the use of a rotational rheometer with coaxial cylinders [19], [32], [33].

Whilst the interactions between cement and plasticizing admixtures have been explored before, not enough scientific data have been found on, in particular, the effectiveness of the plasticizing admixtures and their ability to retain the duration of the plasticizing effect of the cement pastes evaluated by rheological tests. Through our testing process, we discovered and henceforth provide more scientific data on the impact of dosage and effectiveness of commonly used plasticizing admixtures (ligno-sulphonates, modified acrylic polymers and synthetic polycarboxylate esters-based ones), more specifically, on rheological behavior of the cement pastes (flow curves, yield stresses and plastic viscosities) of different testing times (from 0 to 90 min after mixing).

II. RESEARCH METHODOLOGY AND MATERIALS USED

Portland cement CEM I R, according to EN 197-1 (strength class 42.5 R), produced by JSC Akmenes Cementas was used for the purposes of this experimental research. Clinker mineral composition (according to Bogue calculation) was $C_3S - 61.0\%$, $C_2S - 13.5\%$, $C_3A - 8.5\%$, $C_4AF - 10.5\%$, $SO_3 - 3.10\%$, $LOI - 1.43\%$, with particle density being 3.11 g/cm^3 and dry bulk density – 1.22 g/cm^3 . The average size of particle is $15.05\text{ }\mu\text{m}$. 50 % of particles are smaller than $9.94\text{ }\mu\text{m}$. Physical properties of the cement used are displayed in Table I.

The particle size distribution of cement and its granulometry curve are presented in Fig. 1. These parameters were determined using Cilas 1090 Liquid (France). The following plasticizing admixtures were used in the investigation: plasticizer (P), superplasticizers (SP1 and SP2). Characteristics of chemical admixtures are shown in Table II. It is worth mentioning that different values of dry solids content of different plasticizing admixtures were taken into account when determining admixture dosages value in paste mixtures. Chemical admixtures content in cement pastes varied from 0 % to 1.2 % of the cement weight with interval-based addition every 0.2 %. Bigger dosages were not investigated because segregation of cement paste occurred.

Cement paste was mixed in a planetary mixer according to EN 196-1. The mixing procedure was as follows: cement and 3/4 of the required water were mixed for 2 min at high speed. Subsequently, the remaining water and plasticizing admixture (P, SP1 or SP2) were added. Finally, cement paste was mixed further for 3 min at high speed.

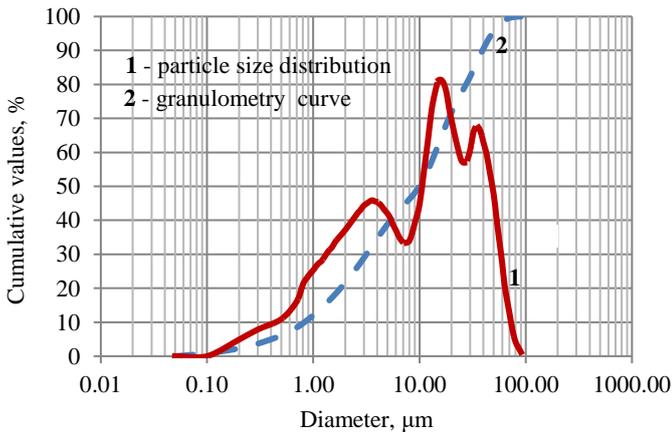


Fig. 1. Particle size distribution of cement CEM I 42.5 R and its granulometry curve.

TABLE I
PHYSICAL PROPERTIES OF THE CEMENT (ACCORDING TO EN 197-1)

Type of cement	Compressive strength, MPa		Setting time, min		Standard consistence (water content in, %)	Fineness	
	7 days	28 days	initial	final		Blaine, cm^2/g	$> 90\text{ }\mu\text{m}$, %
CEM I 42.5 R	28.9	54.6	150	200	25.4	3560	1.1

TABLE II
TECHNICAL INFORMATION ON CHEMICAL ADMIXTURES

Class of admixture	P	SP1	SP2
Type of admixture	Plasticizer on the basis of ligno-sulphonates	Superplasticizer on the basis of modified acrylic polymers	Superplasticizer on the basis of synthetic polycarboxylate ester
pH (20 °C)	8.00 ± 1.00	6.50 ± 1.00	6.00 ± 1.00
Bulk density, g/cm ³	1.20 ± 0.03	1.06 ± 0.02	1.06 ± 0.01
Max. chloride content, %	≤ 0.05	< 0.05	≤ 0.10
Max. alkali content, %	≤ 6.00	< 2.50	≤ 0.10
Dry solids content, %	39.00 ± 2.00	22.00 ± 1.00	27.00 ± 1.00

The rheological properties of cement paste were tested using rotational rheometer (Rheotest RN4) with coaxial cylinders with the gap between cylinders of 1.48 mm.

The cylinder measuring system consists of the measuring cup (assembled) (1) with coupling (3) and cylinder rotor (2). The dimensions of testing cylinders are presented in Fig. 2.

The cement paste is poured into a measuring cup (1), which is fixed and stationed in the equipment stand. Inside the measuring cup the cylinder rotor (2) rotates. Because of the intrinsic friction of the layers of the cement paste (4) observed between the measuring cup (1) and the rotating cylinder rotor (2) positioned

in the measuring cup and connected to a measuring scale, the cylinder rotor (2) makes a turn and the data displayed on the measuring scale changes.

The shear rates used in the test are presented in Fig. 3.

Rheological properties (yield stress τ_0 , plastic viscosity $\mu_{pl.}$) of the cement paste were tested at different times after paste mixing: 5 min, 15 min, 30 min, 45 min, 60 min, 80 min and 90 min.

The cement paste (W/C = 0.32) with plasticizer P and paste (W/C = 0.30) with superplasticizer SP1 or SP2 were tested. A test of rheology was carried out at temperature (20 ± 2) °C.

The yield stress (τ_0) and plastic viscosity ($\mu_{pl.}$) of the cement paste were calculated based on the flow curve (shear stress “ τ ” and shear rate “ γ ” curve) obtained after the test with rotational rheometer (Fig. 3 “II”). The flow curve of the cement paste was analyzed adopting the Bingham rheological model using linear approximation of experimental results up to 100 s⁻¹ shear rate when the flow curve of cement paste is linear (Fig. 4).

The yield stress of the cement paste (τ_0) and plastic viscosity ($\mu_{pl.}$) values were obtained from linear equation (Bingham rheological model):

$$\tau = \tau_0 + \mu_{pl.} \cdot \gamma,$$

where: τ – shear stress, Pa;

τ_0 – yield stress of the cement paste, Pa;

$\mu_{pl.}$ – plastic viscosity of the cement paste, Pa·s;

γ – shear rate, s⁻¹.

The rheological properties of the cement paste gradually change with time due to cement hydration process.

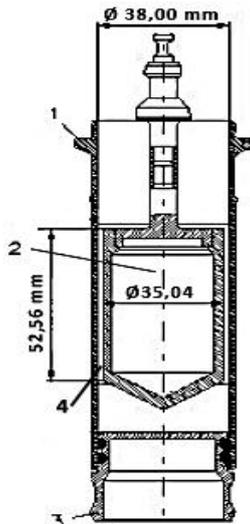


Fig. 2. Principle schema of the testing cylinders.

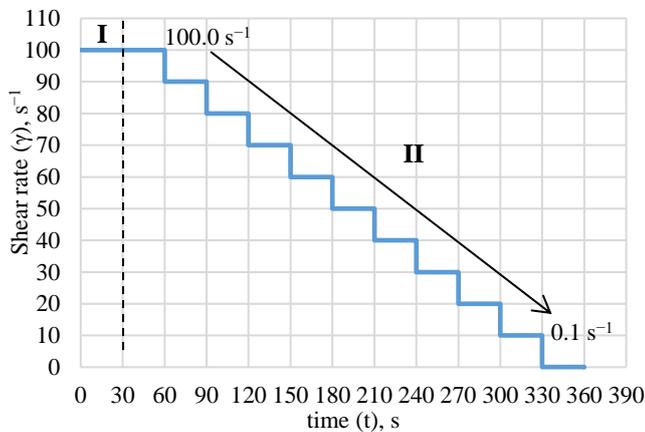


Fig. 3. Shear rates during testing vs. testing time.

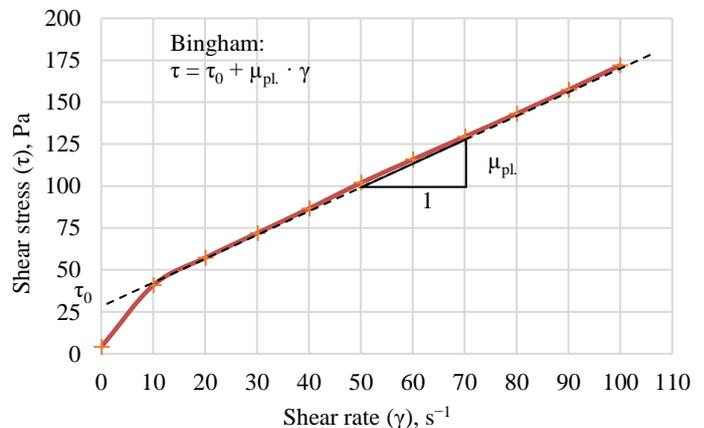


Fig. 4. Calculation of yield stress, viscosity of the cement paste from flow curve.

It appears that effectiveness of some plasticizing admixtures is limited in duration. The effectiveness of plasticizing admixtures is calculated using the following equation:

$$K_{\text{eff}} = \frac{\mu_{\text{pl},0} - \mu_{\text{pl},i}}{\mu_{\text{pl},0}} \cdot 100, \%$$

where: $\mu_{\text{pl},i}$ – plastic viscosity of the plasticized cement paste, Pa·s;

$\mu_{\text{pl},0}$ – plastic viscosity of the cement paste without plasticizing admixture, Pa·s.

III. RESULTS OF EXPERIMENTAL RESEARCH

A. Flow Behaviour of Plasticized Cement Pastes

From the determined flow curves (Fig. 5–7), 5 min after the start of mixing, it is clear that cement pastes with different types of plasticizing admixtures exhibit different rheological properties.

From Fig. 5 we can see that having mixed plasticizer P into the cement paste (dosage of P increase is 0.0–1.2 % of the cement weight, in this case), the rheological properties of the paste improve, as shear rate is increasing from 0 s⁻¹ to 100 s⁻¹. Lesser shear stress is observed, compared to cement paste without P.

5 min after mixing of the paste, we can see that having mixed superplasticizer SP1 or SP2 (SP1 or SP2 is 0.0–1.2 % of the cement weight, in this case) into the cement paste, the rheological properties of the paste improve: while the shear rate is increasing

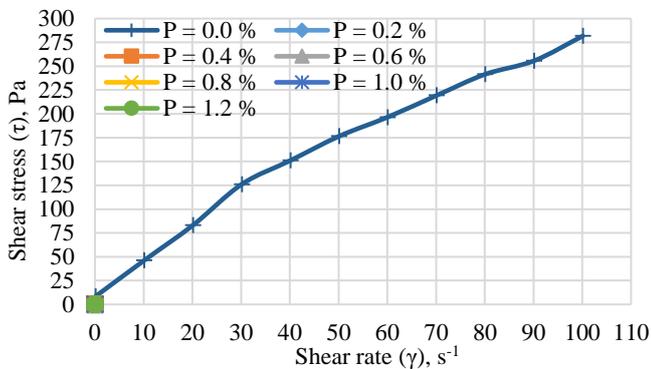


Fig. 5. Flow curves of cement pastes with plasticizer P 5 min after mixing of the pastes.

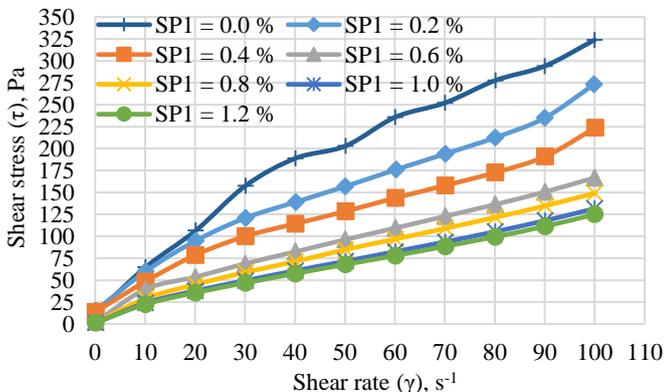


Fig. 6. Flow curves of cement pastes with superplasticizer SP1 5 min after mixing of the pastes.

from 0 s⁻¹ to 100 s⁻¹, lower shear stress is observed compared to cement paste without SP1 or SP2 (Fig. 6 and Fig. 7).

We can conclude that superplasticizer SP2 (based on polycarboxylate ester) has a better plasticizing effect on the paste, given the same composition (W/C = 0.30), compared to superplasticizer SP1 (based on modified acrylic polymers). Having increased the dosage of plasticizing admixture from 0 % to 0.8 %, in the range of shear rate from 0 s⁻¹ to 100 s⁻¹, shear stresses decreased: with SP1 – from 324 Pa to 149 Pa, with SP2 – from 324 Pa to 104 Pa, 5 min after mixing of the paste.

Therefore, as from 0.8 % to 1.2 % the decrease of shear stresses of cement pastes is marginal. 0.8 % could be considered the recommended dosage.

B. Yield Stresses of Plasticized Cement Pastes

In Fig. 8 we can see that having mixed from 0.0 to 0.4 % of plasticizer P into the cement paste, paste yield stresses are decreasing from 29.75 Pa to 18.14 Pa, while having mixed 0.4–1.2 % of P, yield stresses are increasing from 18.14 Pa to 29.43 Pa, 5 min after mixing of the paste.

From the start of mixing of the paste to 15 min margin, the cement paste with P up to 0.4 %, exhibits relatively short duration of plasticizing effect. Notably, in the period from 15 to 90 min intensifying yield stresses are observed. Meanwhile, as we increase the dosage of plasticizer P from 0.4 % to 1.2 %, marginally increasing yield stresses are observed until 80 min, and they increase intensely from 80 min to 90 min (Fig. 8).

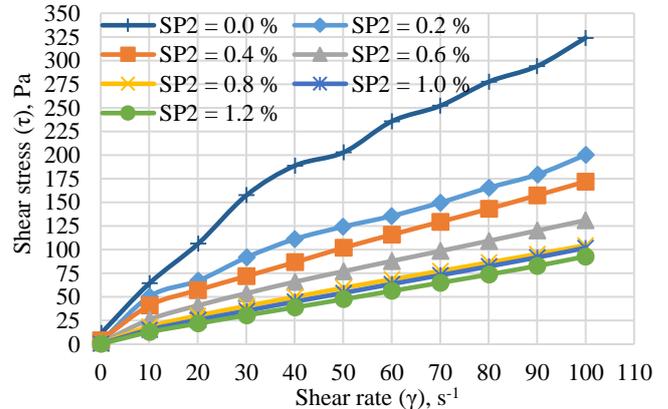


Fig. 7. Flow curves of cement pastes with superplasticizer SP2 5 min after mixing of the pastes.

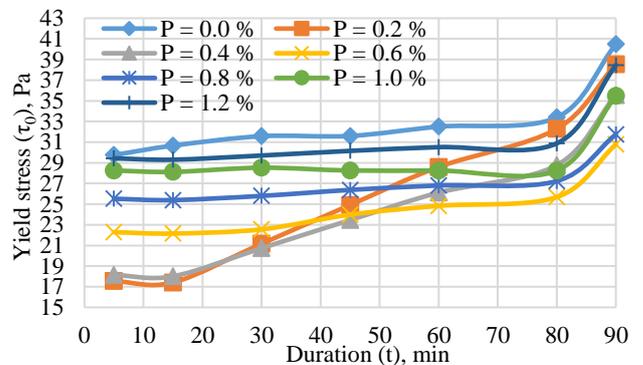


Fig. 8. Change of yield stress of plasticized cement pastes with plasticizer P within 90 min duration.

By increasing the dosage of superplasticizer SP1 or SP2 from 0.0 % to 1.2 %, yield stresses of the cement pastes decrease. In addition, we can see that SP1 or SP2 effectiveness of plasticizing of cement pastes from the start of mixing until 90 min changes: the yield stresses of cement pastes remain constant from the start of mixing until 80 min after. From 80 min to 90 min onwards intensive yield stress increases (Fig. 9 and Fig. 10).

Thus, we can conclude that superplasticizer SP2 is more effective than SP1 – the cement paste exhibits better flowability and improved rheological qualities. Yield stresses of plasticized cement pastes with SP1 or SP2 decrease depending on admixture dosage (0.0–1.2 %): with SP1 – from 45.23 Pa to 8.98 Pa and from 58.57 Pa to 12.00 Pa, with SP2 – from 45.23 Pa to 2.73 Pa and from 58.57 Pa to 6.19 Pa, 5 min and 90 min after the start of mixing, respectively.

C. Plastic Viscosities of Plasticized Cement Pastes

As the dosage of plasticizer P is increased from 0.0 to 1.2 %, plastic viscosity of the cement paste decreases.

In Fig. 11 we can see that in the cement paste with plasticizer P plastic viscosity changes marginally from the start of mixing to until 80 min, from 80 min to 90 min intensive increase is observed. In addition, we can see that the cement paste with SP1 or SP2 plastic viscosity marginally increases from the start of mixing until 80 min after. From 80 min to 90 min onwards intensive plastic viscosity increases (Fig. 12 and Fig. 13).

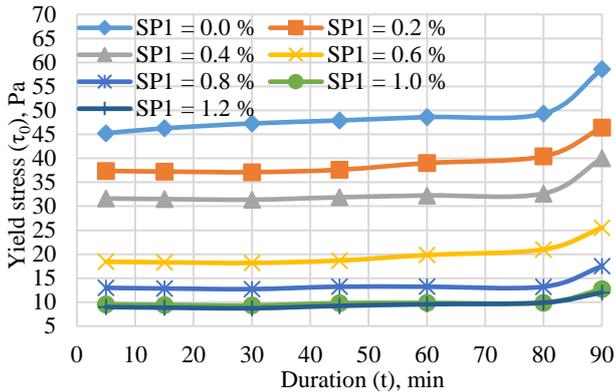


Fig. 9. Change of yield stress of plasticized cement pastes with superplasticizer SP1 within 90 min duration.

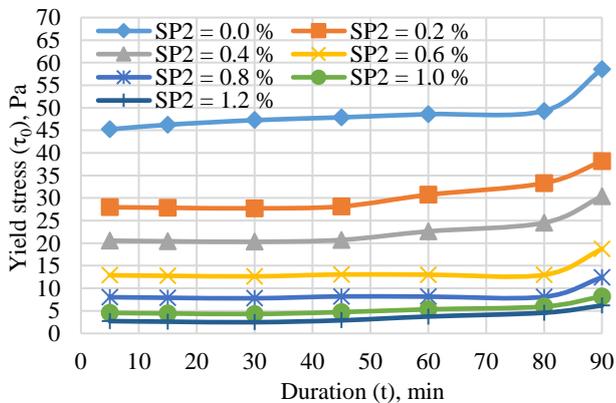


Fig. 10. Change of yield stress of plasticized cement pastes with superplasticizer SP2 within 90 min duration.

Thus, we can conclude that superplasticizer SP2 is more effective than SP1 – the cement paste exhibits better flowability and improved rheological qualities. Plastic viscosity of plasticized cement pastes with SP1 or SP2 changes within dosage from 0.0 % to 1.2 %: with SP1 – from 2.94 to 1.15 Pa·s and from 3.66 to 0.95 Pa·s, with SP2 – from 2.94 to 0.89 Pa·s and from 3.66 to 0.95 Pa·s, 5 and 90 min after the start of mixing, respectively.

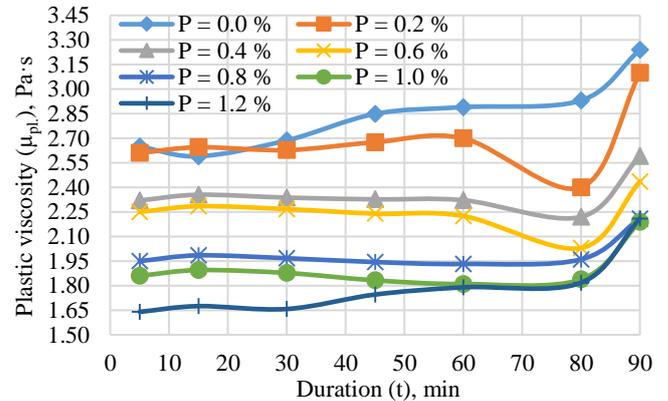


Fig. 11. Change of plastic viscosity of plasticized cement pastes with plasticizer P within 90 min duration.

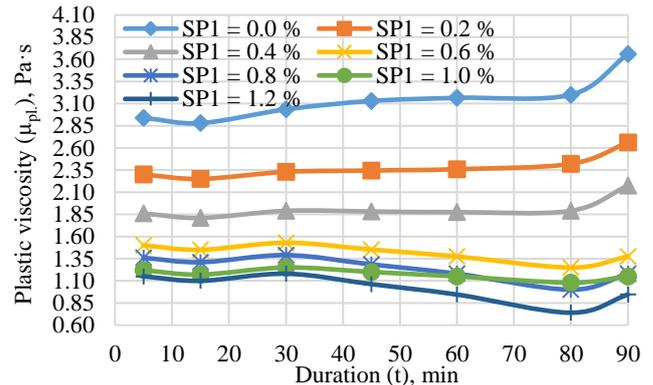


Fig. 12. Change of plastic viscosity of plasticized cement pastes with superplasticizer SP1 within 90 min duration.

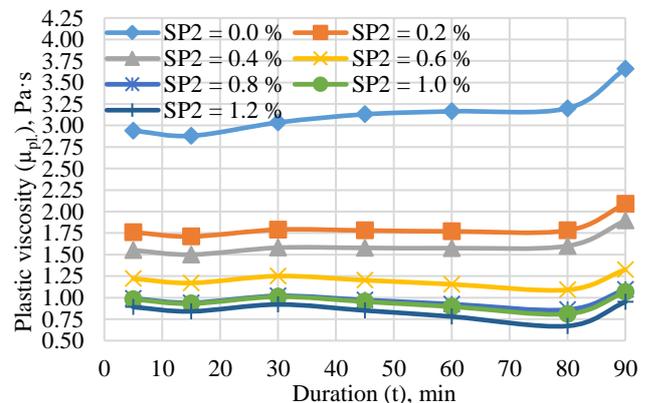


Fig. 13. Change of plastic viscosity of plasticized cement pastes with superplasticizer SP2 within 90 min duration.

D. Effectiveness of Plasticizing Admixtures in Cement Pastes

In Fig. 14 we can see that different types of plasticizing admixtures exhibit different levels of plasticizing effectiveness and ability to retain the duration of the plasticizing effect.

As we increased the dosage of P from 0 % to 1.2 % of the cement paste, the plasticizing effect increased by 11–44 % and 3–31%, 5 and 90 min after the start of mixing, respectively. Best plasticizing effect and effect retention have been reached when the dosage of P is 0.8 % (Fig. 14a). Greater dosages of P (0.8–1.2 %) increase the plasticizing effect of the paste, but have no effect on the duration of the effect.

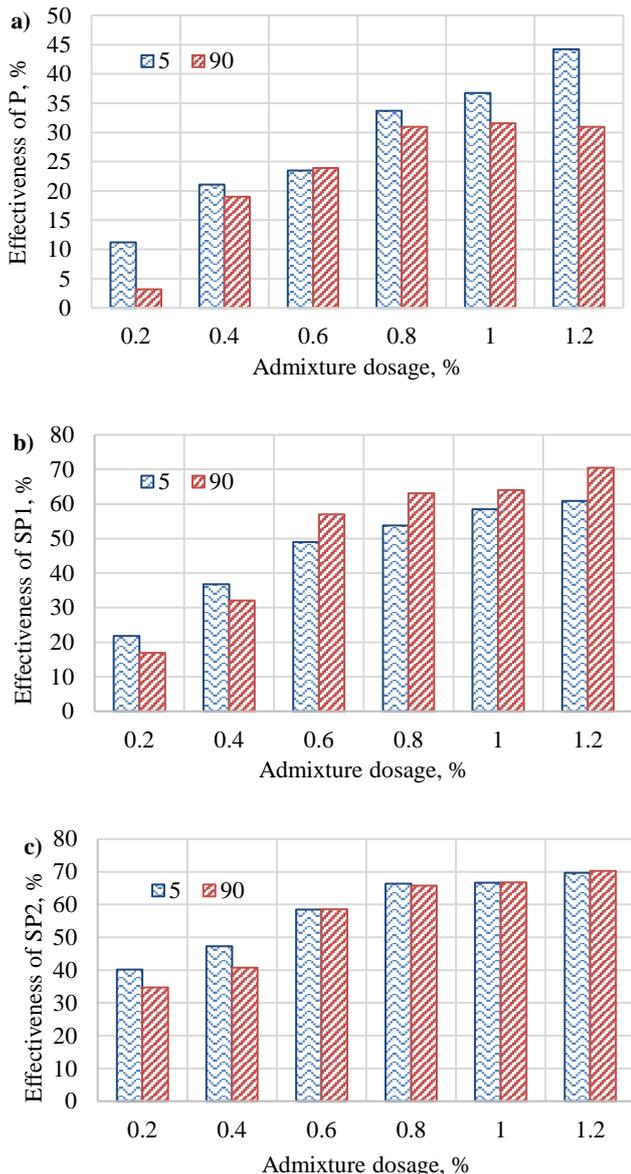


Fig. 14. The changes of effectiveness of (a) plasticizer P, (b) superplasticizer SP1 and (c) superplasticizer SP2 in cement pastes 5 and 90 min after mixing of the pastes.

Due to the increase in the dosage of superplasticizers from 0.0 % to 1.2 %, plasticizing effectiveness changes: with SP1 22–61 % and 17–70 %, with SP2 40–70 % and 35–70 %, after 5 min and 90 min from the start of mixing, respectively. It is also observed that larger dosage of SP1 (0.6–1.2 %) results in slower

increase in plasticizing effects until the 90 min margin. Delayed fluidification can be observed with acrylic backbones because they rapidly lose their side chains through hydrolysis at high pH [33]. Their charges increase over time, as well as their dispersion efficiency (at least as long as enough side chains remain on the polymer). Recommended SP1 dosage range is from 0.6 to 0.8 %.

In conclusion, from the start of mixing until the 90 min margin, the best plasticizing effect and its retention have been achieved by superplasticizer SP2 (polycarboxylate ester based). Recommended SP2 dosage range is from 0.6 to 0.8 %.

IV. CONCLUSION

This work has described research of 3 different plasticizing admixtures, their effectiveness and ability to retain the duration of the plasticizing effect. We sought to test an elaborate evaluation methodology of plasticizer effects on cement pastes. This research produced the following findings:

1. Having mixed 0.0 % to 0.4 % of plasticizer P (ligno-sulphonates based) into the cement paste ($W/C = 0.32$), paste yield stress decreases, while having mixed from 0.4 % to 1.2 % of P, yield stress increases.
2. As the dosage of plasticizer P (ligno-sulphonates based) is increased from 0.0 % to 1.2 %, plastic viscosity of the cement paste decreases.
3. As the dosage of superplasticizer SP1 (modified acrylic polymer based) or SP2 (polycarboxylate ester based) is increased from 0.0 % to 1.2 %, yield stress and plastic viscosity of the cement paste ($W/C = 0.30$) decrease. Superplasticizer SP2 is more effective than SP1: cement paste with this admixture exhibits better flowability and improved rheological qualities.
4. Different types of plasticizing admixtures exhibit different levels of plasticizing effectiveness and respective ability to retain the duration of the plasticizing effect. Best plasticizing effect and effect retention were reached when the dosage of plasticizer P (ligno-sulphonates based) was 0.8 %. Greater dosages of P (0.8–1.2 %) increase the plasticizing effect of the paste, but have no effect on the duration of the plasticizing effect.
5. Due to the increase in the dosage of superplasticizers SP1 (modified acrylic polymer based) and SP2 (polycarboxylate ester based) from 0.0 % to 1.2 %, plasticizing effect increased. It has also been observed that larger dosage of SP1 (0.6–1.2 %) results in slower increase in plasticizing effects until the 90 min margin. In conclusion, from the start of mixing until the 90 min margin, the best plasticizing effect and its retention have been achieved by superplasticizer SP2. Recommended SP2 dosage is from 0.6 to 0.8 %.

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