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Observation of nano powders and fly ash usage effects on the fluidity features of grouts

Fatih Celik*[1], Oguzhan Yıldız[2a] and Samet M. Bozkır[1b]

[1]*Department of Civil Engineering, Nigde Omer Halisdemir University, Nigde, Turkey*
[2]*Department of Electricity and Energy, Nigde Technical Vocational School, Nigde Omer Halisdemir University, Nigde, Turkey*

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Abstract. The pumpability of the grouts is significant issue in concept of the rheological and workability properties during penetrating to voids and cracks. To improve the fluidity features of the grout mixes, the usage of Colloidal Nano Particular Powders (CNPPs) with mineral additives such as fly ash (FA) can contribute. Therefore, the main purpose of this study can be explained as investigating the usage effects of four types of Colloidal Nano Particular Powders (n-TiO_2, n-ZnO, n-Al_2O_3 and n-SiO_2) as nano additives on the rheological, workability and bleeding properties of cement-based grout incorporated with fly as. Test results showed that the usage of FA in the grout samples positively contribute to increase on the fluidity of the grout samples as expected. The dilatant behavior was observed from the results for all mixes. Observing the effect of nano-sized additives in such cement-based grout mixtures with high fluidity has presented remarkable effects in this study.

Keywords: carbon dioxide trapping; mathematical modelling; stability analysis; trapping material mechanism

1. Introduction

The pumpability of the grouts is significant issue in concept of the rheological and workability properties during penetrating to voids and cracks. Therefore, increasing the fluidity of such grouts without adversely affecting their mechanical properties will both contribute to an easy pumping process and help these mixtures to leak and spread easily on the soils. Generally, water to binder ratio (w/b) for cement-based grouts is known as dominant factor to enhance the flow capacity of grout because its viscosity can be decreased by increasing the total water amount in the mixtures. Therefore, different w/b ratios at a wide range are used during applying grout injection methods. Whereas w/b ratio for the coating of prestressed cables is changed usually between 0.35 and 0.42, this ratio changes between 0.5 and 1.5 for the repair and consolidation of masonry structures. Moreover, this ratio is usually used between 0.6 and 2 for jet grouting applications in geotechnical works, and it also selected between 0.5 and 1 for sealing cement grouts (Woodward and Miller 1990, Moseley 1993, Cry *et al*. 2000).

However, high w/b ratios usually cause several adversely effects on cement-based grouts especially in mechanical properties. The rate of bleeding of the fresh grout increases because of increase of w/b ratio, and most mechanical features of grouts after hardening decreases. Therefore, some supplementary cementitious materials (SCMs) as mineral additives such as fly ash, silica fume, ground granulated blast furnace slag, metakaolin, bentonite and rice husk ash are generally used to control adversely effects of high amount of water in the grout sets. Generally, many physical and chemical properties of the fresh and hardened grouts such as the heat of hydration, the resistance against chemical attack and the overall durability can be improved by incorporation of most SCMs (Sonebi 2006, 2010, 2013, Yamamoto and Kobayashi 1986, Golaszewki and Szwabowski 2004, Jolicoeur *et al*. 1997, Petit *et al*. 2005, 2009). To satisfy required better physical features of grouts such as rheological, mechanical, and durability, using these mineral additives and selecting the appropriate mix design is accepted as a critical issue (Sonebi *et al*. 2014). There are a few of mineral additives like fly ash that improve not only durability features but also enhance the rheology, workability, and long-term performance of grouts (Sonebi 2002). By incorporation of most SCMs, the cost of grouting applications can be reduced while the fluidity and the long-term capacity of cement-based grouts can be increased.

In recent years, the usage of Colloidal Nano Particular Powders (CNPPs) as additive in cement-based grouts has started to be popular with or without mineral additives. Several features of cement-based grouts can be improved by incorporation of these nano powders (Peng *et al*. 2019). Not only mechanical features of cement-based grouts can be improved, but also rheological and workability properties can be enhanced by using CNPPs as additive (Rashad 2013, Song *et al*. 2018). Some recent works have shown that the setting time of fresh grouts can be reduced by the addition of nano silica (n-SiO2) while it can attribute to generate more hydration products in cement-based grouts (Singh *et al*. 2012, Madani *et al*. 2012, Bjornstrom *et al*. 2004). Moreover, the pore structure of concrete may be improved

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*Corresponding author, Associate Professor,
E-mail: fatihcelik@ohu.edu.tr
[a]Ph.D., E-mail: oguzhan.yildiz@ohu.edu.tr
[b]M.Sc. Student, E-mail:
sametmufit_bozkır@mail.ohu.edu.tr

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by addition of nano silica (n-SiO2) (Nazari and Riahi 2011, Hou *et al*. 2013). As it is known from the past studies the water requirement of the cement-based matrix shows an increase with rising of nano silica content because of having large specific surface area (Zabihi and Ozkul 2018, Balapour *et al*. 2018, Ashok *et al*. 2017). On the other hand, the prediction of the minimum apparent viscosity obtained from the cement-based grouts prepared by addition of nano silica was studied at different w/b ratios (Ouyang 2018), and an analytical prediction model for viscosity determination was presented in this study. Another CNPPs used in cement-based grouts as an additive is nano alumina (n-Al2O3). The past investigations have showed that specific surface area of nano alumina is very close to nano silica because of both having similar nano colloidal structure (Ouyang *et al*. 2018, Oltulu and Sahin 2013). Higher ettringite contents can be observed by the initial dissolution of nano alumina at the early hydration process. One of CNPPs is nano calcium carbonate (n-CaCO3) that was investigated for cement-based grouts as an additive (Liu *et al*. 2012). According to this study, this nano powder has a little effect on the arranging of required water amount for cement-based grouts. However, this study showed that an increase of n-CaCO3 amount in cement paste matrix by weight up to 2% causes a slightly reduction on fluidity of the fresh grout, and also the setting time of the grout can be shortened.

There are several past studies that were focused on the rheological properties of cement-based grouts prepared by addition of CNPPs (Collepardi *et al*. 2005, Senff *et al*. 2009). Collepardi *et al*. (2005) investigated the effect of the nano silica usage as an additive on the cement-based grout. They found that increase of nano silica amount in cement matrix causes a reduction on the setting time, segregation, and bleeding, and also can improve the cohesion of the grout mix. Moreover, Senff *et al*. (2009) investigated the rheological properties of cement-based grouts prepared by addition of nano silica at different amount between 0% and % 2,5% by mass of binder amount at constant water to binder ratio (w/b=0.35). They found from their study that the yield stress (cohesion) of the fresh grout samples shows considerable rising with increase of nano silica amount. On the other hand, they have presented that increase of nano silica amount in cement-based grouts makes viscosity of fresh mix increase. These recent studies have showed that the usage of CNPPs as additive in cement-based grouts has remarkable effects on the rheological and fluidity features of fresh grout matrixes. However, generally one type of CNPPs like especially nano silica was used in these past studies. Moreover, any study based on addition of CNPPs to fresh grout mix has not been conducted on rheological properties of cement-based grouts incorporated with fly ash as mineral additive.

As it can be understood from these past studies, the use of mineral additives and nano powders as additives in grout mixtures reduced the negative effects of high w/b ratio and provided remarkable contributions on the fluidity properties of these mixtures. Considering these positive contributions of both additives (CNPP and mineral additive), it was evaluated how the use of them together would influence the fluidity properties of grout mixtures, and within the scope of this study, the use of four different CNPPs with fly ash was investigated. Therefore, the effects of four different types of Colloidal Nano Particular Powders (CNPPs) (n-SiO_2, n-TiO_2, n-ZnO and n-Al_2O_3) additions at different amounts by mass (0.0%, 0.3%, 0.6%, 0.9%, 1.2% and 1.5%) on rheological, workability and stability of cement-based grouts incorporated with fly ash as mineral additive at different constitutes (%0-for control purpose, 5%, 10%, 15%, 20%, 25% and 30%) were investigated in this study.

Table 1 Some physical and chemical properties of Portland Cement (PC) and FA

Chemical Analysis (%)	CEM-I 42.5 R (Cement)	Fly ash (FA)
CaO	60.15	1.72
SiO_2	20.46	62.21
Al_2O_3	7.78	21.15
Fe_2O_3	3.09	7.29
MgO	2.66	1.59
SO_3	2.33	0.15
K_2O	0.82	2.12
Na_2O	0.22	0.94
TiO_2	0.30	0.83
Physical Analysis		
Loss on ignition	2.55	2.01
Specific gravity	3.10	2.33

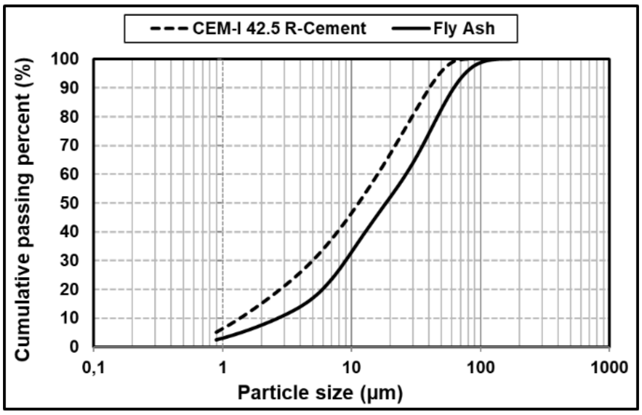
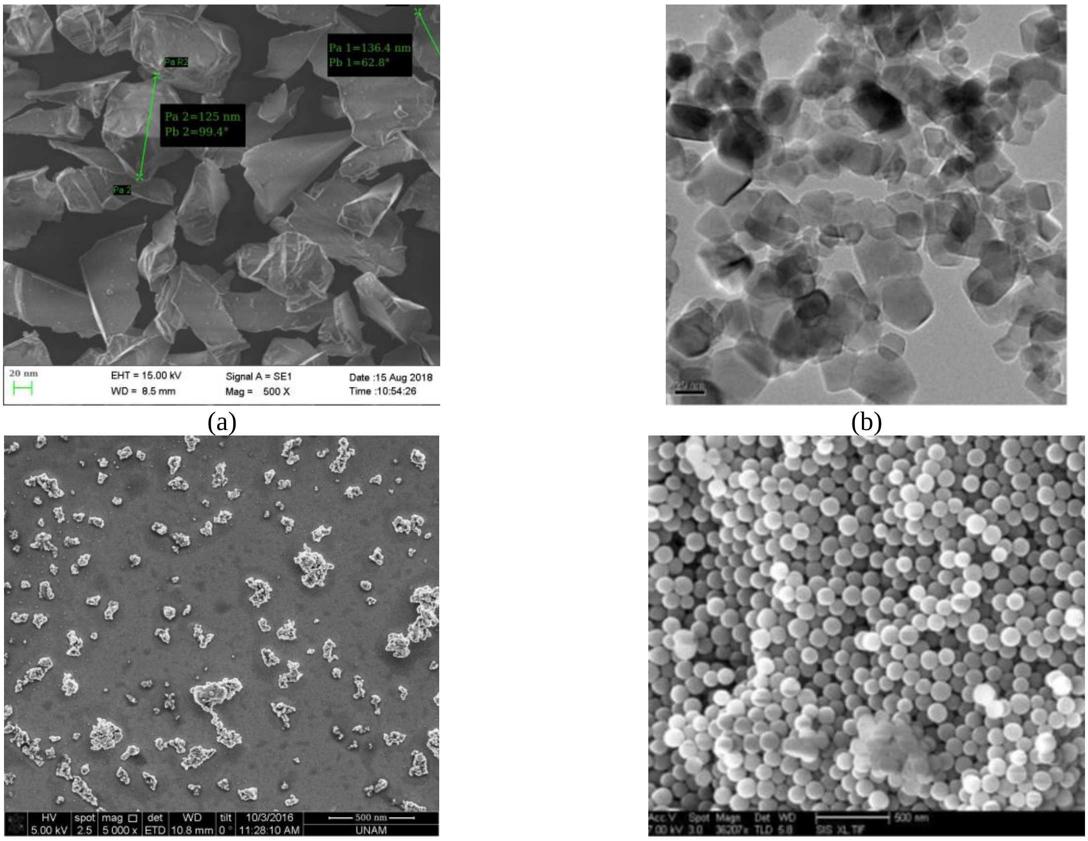


Fig. 1 The particle size distribution curves of Portland cement and Fly ash (FA)

2. Experimental program

2.1 Materials used in this study

CEM I-42.5R (Type-I) Portland cement (PC) based on ASTM C150 was used in this study. To produce the cement-based grout mixes with mineral additive, F-Class fly ash (FA) that was obtained from the Isken Sugozu Termal Plant at Iskenderun-Turkey was used as mineral additive in this work. Some chemical and physical properties of cement and FA were given in Table 1. All properties given in Table 1 were obtained from the companies that supplied the cement and FA used in this



(a) (b)

(c) (d)

Fig. 2 The SEM views of CNPPs as nano additive used in this study

Table 2 Elemental analysis results for CNPPs used in this study

Elemental Analysis	Na	Fe	Cr	Ca	Mn	Co	K	Al	Cu
Nano Alumina (n-Al_2O_3) (ppm)	<70	<80	<4	<25	<3	<2	-	-	-
Nano Titanium Oxide (n-TiO_2) (%)	0.0076	0.0046	-	-	-	-	0.0085	0.0055	-
Nano Silica (SiO_2) (%)	-	0.0056	-	0.022	-	-	-	-	-
Nano Zinc Oxide (n-ZnO) (%)	-	0.001	-	0.002	0.0005	-	-	0.002	0.0003

study. One of the important parameters on the rheological features of cement-based grouts is particle size distribution of the binders (cement and mineral additives). Therefore, the particle size distribution curves of the cement and FA used in this study are shown in Fig. 1. As it is clearly seen from Fig. 1, the particle size distribution of FA is very close to the cement used in the study. Almost all particle size of FA is less than 100 microns like cement. As it is understood from the results, this particle size gradation of FA has contributed to the rheological properties of cement-based grouts. These curves were plotted by using laser scattering technique with Master-sizer machine.

In this experimental work, nano Alumina (n-Al_2O_3), nano Silica (n-SiO_2), nano Titanium Oxide (n-TiO_2) and Nano Zinc Oxide (n-ZnO) were used as CNPPs. The SEM views of all CNPPs used as nano additive in this work are shown in Fig. 2. All these CNPPs were obtained by a commercial firm called as Nanografi in Turkey. Some physical and chemical properties of these CNPPs are seen in Tables 2 and 3.

2.2 Preparing the samples and mix design

As it has been mentioned before, one of the most important parameters that directly influences the mechanical, durability and rheological properties of cement-based grouts is water to binder ratio (w/b). In this study w/b ratio was defined as one. This selected value is widely used in especially for jet grouting and permeation grouting works in geotechnical applications. The effects of four different types of Colloidal Nano Particular Powders (CNPPs) (n-SiO_2, n-TiO_2, n-ZnO and n-Al_2O_3) additions at different amounts by mass (0.0%, 0.3%, 0.6%, 0.9%, 1.2% and 1.5%) on rheological, workability and stability of cement-based grouts incorporated with fly ash as mineral additive at different constitutes (0%-for control purpose, 5%, 10%, 15%, 20%, 25% and 30%) were investigated in this study. All mix ratios and mix design parameters used for preparing the grout samples with cement, FA and CNPPs in this study are presented in Table 4. As it is seen from Table 4, total number of 147 (35x4 with CNPPs and 7 for control

Table 3 Some physical properties of CNPPs used in this work

Physical properties	n-Al_2O_3	n-TiO_2	n-SiO_2	n-ZnO
Purity (%)	99.5	99.6	99.7	99.5
Color	White	White	White	Milk white
Average Particle Size (nm)	18	38	13-23	30-50
Specific Surface Area (m^2/g)	140	35	650	70
Mass density (g/cm^3)	-	0.4	-	-
Specific Heat Capacity (J/Kg. K)	890	-	-	-
Density (kg/m^3)	3900	4100	2200	5500
Weight Loss on Drying (%)	-	1.2	-	-
Loss on ignition (%)	-	3.2	-	-
pH	-	5.5-6.5	-	-
Morphologic Structure	Roughly spherical	-	Porous	-

purpose) grout mixtures were prepared and tested.

There are important constraints of using nanomaterials in cement-based grout preparations. One of these constraints is the lack of effective dispersion method (Meng and Khayat 2018). The more dispersion condition of nanomaterials in cement-based grouts is suitable, the more the usage of these nanomaterials is effective and efficient (Metaxa *et al*. 2010, Kirgiz 2015). To control agglomeration and to provide obtaining more uniform dispersion of nanomaterials in aqueous grout matrix, Ultra-sonification method is usually used (Konsta-Gdoutos *et al*. 2010, Peyvandi *et al*. 2013). Therefore, to prevent precipitation of CNPPs in free water and to supply homogenic distribution of CNPPs in water with help of removing of these nano powders from each other, Ultra-sonification method was applied by using an Ultrasonicator in this study. Ultra-sonification has provided that CNPPs are suspended in water. The view of the Ultrasonicator used in this study is shown in Fig.3. During preparing all fresh grout mixtures, CNPPs were added to volume of 200 ml water, and then Ultra-sonification method was applied to this aqueous media by the Ultrasonicator throughout 180 minutes in this study. After distillation process was completed, this aqueous mixture was poured to the cement-based grout mixtures. Thereby, CNPPs have been able to add to the grout matrixes without any agglomeration and non-uniform dispersion.

Similar mixing methodology was applied to all grout mix during preparing all mixtures. A standard rotary laboratory mixer that has 5-liter volume capacity was used for mixing the samples. To prepare the grout mix, first FA and cement as binders were mixed with water for 1 minute at 240 rpm. Later, mixer was stopped and then both binders were mixed for 1 minute by hand to obtain more homogenous mix. And then, the samples were also mixed again with the mixer for 3 minutes at 240 rpm. Finally, the aqueous media, including CNPPs and 200-ml water and prepared by Ultra-sonification, were poured to cement-based grout matrix and mixed again with mixer for one minute also. Environment temperature and humidity were kept constant as 23 ± 3°C and %55-%65 respectively during preparing all samples and doing the tests.

2.3 Conducting of all rheological, workability and stability tests

After the fresh grout samples were prepared, viscosity and some workability tests were done at the same time. While marsh cone flow, mini slump diameter and plate cohesion tests were being conducting in concept of workability tests, viscosity values of the grout mixtures were also measured by using Coaxial Rotating Cylinder Rheometer (Brookfield Viscometer DV2T) test machine. The equipment views of viscometer, marsh cone, mini slump cone and plate cohesion are detailly shown in Fig. 4. Average test completing duration for one mix proportion was roughly measured 12 minutes after starting mixing procedure. To investigate the stability of the grout samples, bleeding test was also conducted for all mixtures.

The principle of mini slump test is explained as spreading of aqueous grout matrix on a flat plate. The spreading diameter of the grout mix is called as mini slump diameter. Height, bottom, and upper diameters of mini slump cone are 38 mm, 19 mm, and 57 mm, respectively (Kantro 1980). The marsh cone funnel test used in this study is other significant workability test for cement-based grouts. The main principles of this test can be defined as measuring the elapsed time during flowing of fresh grout mix having a specific volume through this cone. This cone has 1500 ml volume, and its internal orifice diameter is 5 mm (Celik and Canakci 2015, Celik and Akcuru 2020, Cry *et al*. 2000). For all grout samples in this test, this marsh cone was filled by the grout mixes that have 1250 ml volume with blocked flow. As soon as flowing of grout started after bottom outlet opened, the elapsed time began to measure, and total duration was recorded for flowing of the grout sample having 1000 ml volume. To compare the results, the flowing duration of only water was tested, and its total flowing duration for 1000 ml volume was measured as 24 second. The other workability test is Lombardi plate cohesion test conducted in this study. The cohesion that is an important parameter directly related with yield stress of grout mix can be measured by using Lombardi plate cohesion test apparatus. There is no international standard about this test, however it has been used in several past

Table 4 All mix ratios and mix design parameters used for preparing the grout samples

Mix ID	w/b	FA (%)*	PC (g)**	FA (g)	Water (g)	CNPPs (g)***	Mix ID	w/b	FA (%)	PC (g)	FA (g)	Water (g)	CNPPs (g)
M1	1.0	-	1500	-	1500	-	M22	1.0	15	1275	225	1500	27
M2	1.0	-	1500	-	1500	9	M23	1.0	15	1275	225	1500	36
M3	1.0	-	1500	-	1500	18	M24	1.0	15	1275	225	1500	45
M4	1.0	-	1500	-	1500	27	M25	1.0	20	1200	300	1500	-
M5	1.0	-	1500	-	1500	36	M26	1.0	20	1200	300	1500	9
M6	1.0	-	1500	-	1500	45	M27	1.0	20	1200	300	1500	18
M7	1.0	5	1425	75	1500	-	M28	1.0	20	1200	300	1500	27
M8	1.0	5	1425	75	1500	9	M29	1.0	20	1200	300	1500	36
M9	1.0	5	1425	75	1500	18	M30	1.0	20	1200	300	1500	45
M10	1.0	5	1425	75	1500	27	M31	1.0	25	1125	375	1500	-
M11	1.0	5	1425	75	1500	36	M32	1.0	25	1125	375	1500	9
M12	1.0	5	1425	75	1500	45	M33	1.0	25	1125	375	1500	18
M13	1.0	10	1350	150	1500	-	M34	1.0	25	1125	375	1500	27
M14	1.0	10	1350	150	1500	9	M35	1.0	25	1125	375	1500	36
M15	1.0	10	1350	150	1500	18	M36	1.0	25	1125	375	1500	45
M16	1.0	10	1350	150	1500	27	M37	1.0	30	1050	450	1500	-
M17	1.0	10	1350	150	1500	36	M38	1.0	30	1050	450	1500	9
M18	1.0	10	1350	150	1500	45	M39	1.0	30	1050	450	1500	18
M19	1.0	15	1275	225	1500	-	M40	1.0	30	1050	450	1500	27
M20	1.0	15	1275	225	1500	9	M41	1.0	30	1050	450	1500	36
M21	1.0	15	1275	225	1500	18	M42	1.0	30	1050	450	1500	45

*Fly ash, **Portland cement, ***Colloidal nano particular powders

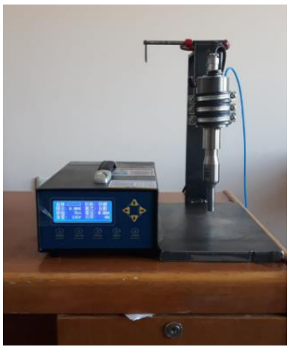


Fig. 3 The view of the Ultrasonicator used in the study

Fig. 4 The view of all workability and rheological tests conducted in the study

(a) at the beginning of the test

(b) at the end of the test after 2 hours

Fig. 5 The bleeding test for stability control

studies for defining the cohesion of cement-based grouts (Celik and Canakci 2015, Celik and Akcuru 2020, Cry *et al*. 2000, Weaver 1991). The principle of this test is based on immersing a metallic plate to fresh grout samples, and measuring the mass of grouts adhered on both side surfaces of the plate that has dimension of 300x300x3 mm.

The stability of cement-based grouts is measured by basic laboratory tests (bleeding test) using 1000 ml grout sample contained in a standard glass cylinder with a uniform diameter of 60 mm (Fig. 5). The volumetric bleeding ($\Delta V/V$), described as the volume of clean water (ΔV) separated from the top of the suspension, is disunited by the original grout volume (i.e., V = 1000 ml) and saved to appraise the stability of a suspension. In previous

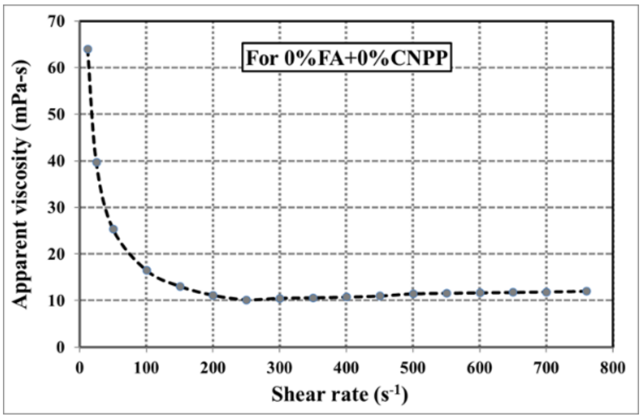


Fig. 6 An example of shear rate versus apparent viscosity curve for one grout sample prepared in this study

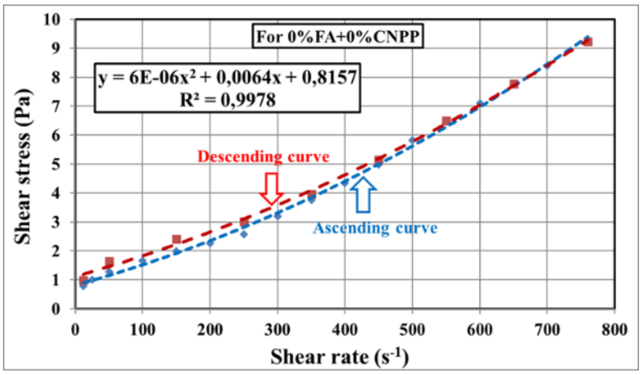


Fig. 7 The showing of shear rate-shear stress curve for ascending and descending conditions to determine Modified Bingham model

studies, if the bleeding rate occurred after 2 hours was between 5% and 10%, it was classified as stable suspension (Kauschınger *et al*. 1992, Celik and Canakci 2018, Kutzner 1974). The high sedimentation rate is characteristic of pure grout and has big practical outcome because if sedimentation of solids consists of during injection, the treated cavities and injection pipelines can become blocked, and the grout cannot flow any further.

All rheological parameters such as apparent viscosity, plastic viscosity and yield stress were measured for the grout samples by using Coaxial Rotating Cylinder Rheometer (Brookfield Viscometer DV2T) in this study. Although the slippage effect was considered in a few past studies (Barnes 1995, Saak *et al*. 2001), this effect was not investigated because of using high shear rates in all experiments conducting for this study. To observe the shear stress and apparent viscosity at wide range, the shear rates were selected between 12 s^{-1} and 760 s^{-1} (see Fig. 6). Application of any pre-sheared method was not to be deemed necessary for this study because of that ascending and descending behaviors were observed during conducting the rheology tests (see Fig. 7).

There are many mathematical models presented in the literature to specify the rheological parameters of grout matrixes, however selecting the most appropriate model is so important to define true flow behavior of grout mixtures (Celik and Canakci 2015, Celik and Akcuru 2020). Although Bingham model is the most usual one to define the non-Newtonian fluids, it may not give reliable results for the samples that show dilatant flow behavior (Yahia and Khayat 2001, Khayat and Yahia 1997). Therefore, Modified Bingham analytical model was used to define the rheological features and flow behavior of the grout samples prepared for this study because of that the samples show dilatant (shear-thickening) flow behavior (as shown in Fig. 7). This mathematical model is expressed by a second order polynomial equation (Khayat and Yahia 1997). This mathematical model is defined as following equation:

$$\tau = \tau_o + \mu_p \dot{\gamma} + c \tag{1}$$

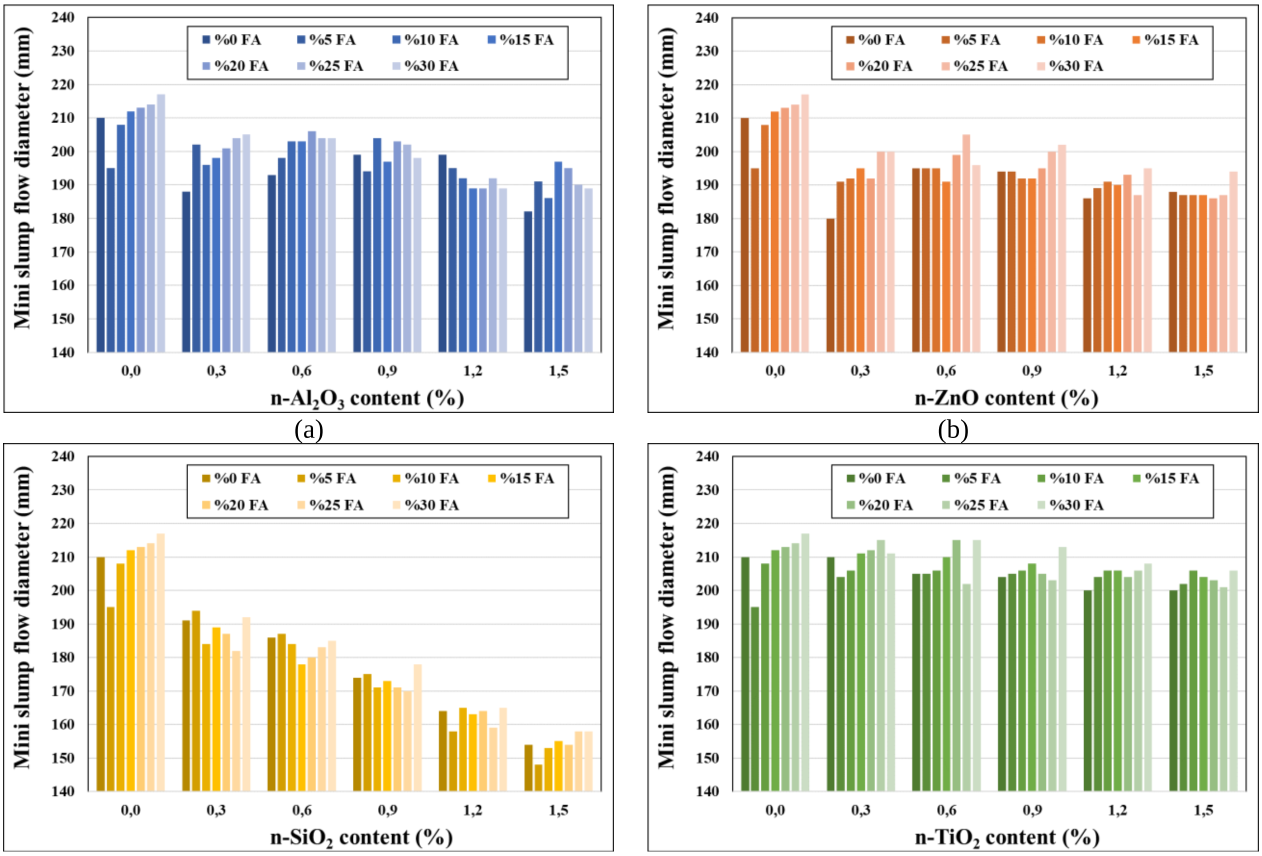
where, τ= shear stress (Pa), τ_0= yield stress (Pa), μ_p= plastic viscosity (Pa.s), $\dot{\gamma}$ = shear rate (1/s) and c=constant.

3. Test results and discussions

3.1 Workability test results of the samples

In concept of this experimental work, the workability features of the cement-based grout mixtures incorporated with FA and CNPPs (n-SiO_2, n-TiO_2, n-ZnO and n-Al_2O_3) were investigated. Marsh cone flow time, mini slump spreading diameter and Lombardi plate cohesion of the grout samples were measured, and all result are presented in Figs. 8-10.

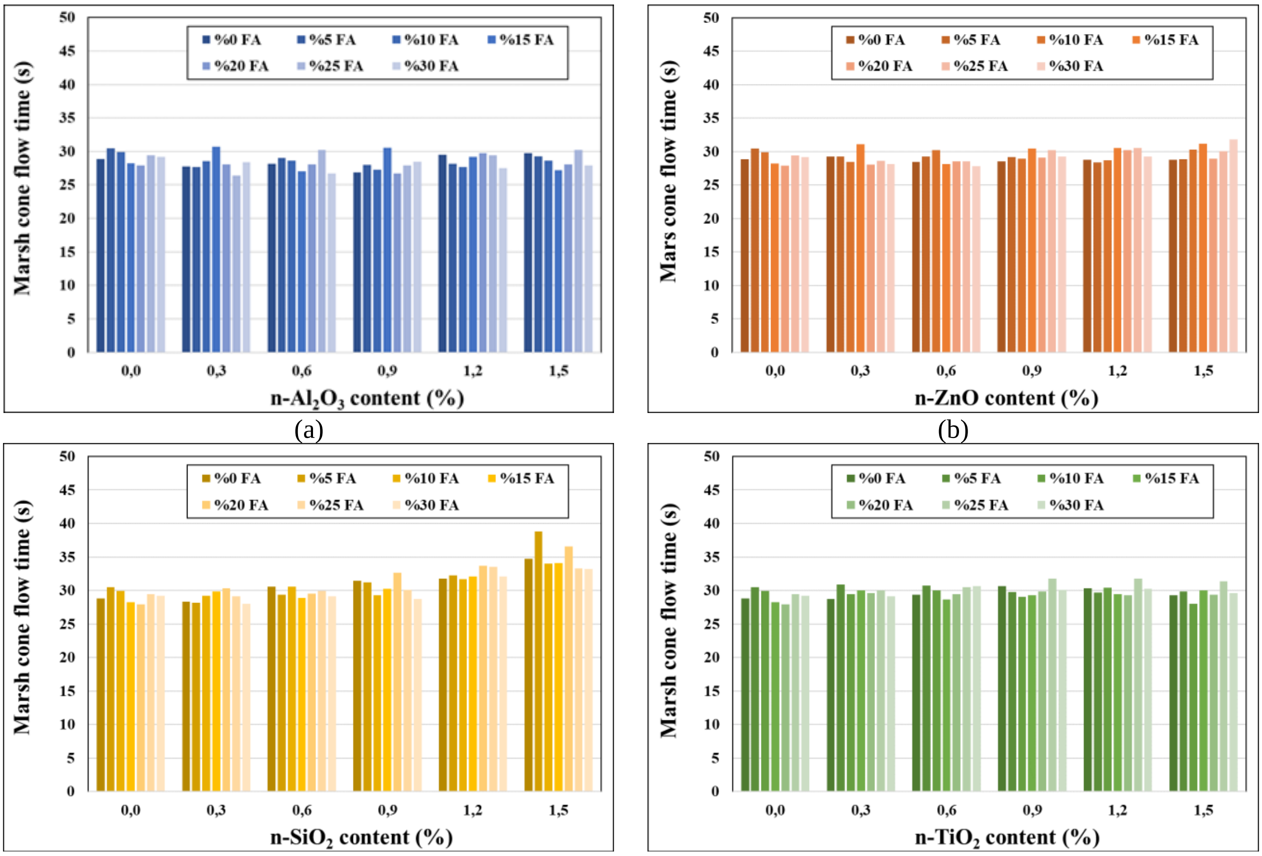
When the results obtained from the samples prepared by addition of n-TiO_2, n-ZnO, n-Al_2O_3 and n-SiO_2 as CNPPs are evaluated for mini slump test, an increase on mini slump spreading diameters can be observed with respect to rising amount of FA in the samples (see Fig. 8). It can be thought that the usage of FA as mineral additive in the grout samples positively contribute to increase on the fluidity of the grout samples as expected (Sonebi 2002). Increase amount of n-TiO_2 in the grout mixtures has made mini slump flow diameter of the samples slightly decrease. However, it cannot be mentioned about considerable reduction as can be seen in Fig. 8. On the contrary to this, according to Fig. 8, this reduction was more pronounced and noticeable in the grout samples prepared with the others CNPPs additives (for n-ZnO, n-Al_2O_3 and n-SiO_2). Especially, the dramatic reductions in the mini slump flow diameters of the grout samples prepared by addition of n-SiO_2 have been observed with respect to increase of nano silica amount in the mixtures. The meaning of the reduction on the mini slump flow diameter of the samples can be explained as increase of cohesion in the sample and decrease of fluidity of the samples at a specific rate (Zabihi and Ozkul 2018, Balapour *et al*. 2018). As can be seen in Table 3, the specific surface areas determined for the CNPPs n-TiO_2, n-ZnO, n-Al_2O_3 and n-SiO_2 were measured as 35, 70, 140 and 650 m^2/g, respectively. According to these values, it is understood that n-SiO_2 has the highest specific surface area in all CNPPs, while n-TiO_2 has the lowest degree. Both the dramatic decrease in the mini slump diameter values of the n-SiO_2 added grout mixes with the increase in the amount of nano material, and the fact that the mini slump diameter values of the n-TiO_2 added grout mixes are not significantly affected by the increase in the



(a) (b)

(c) (d)

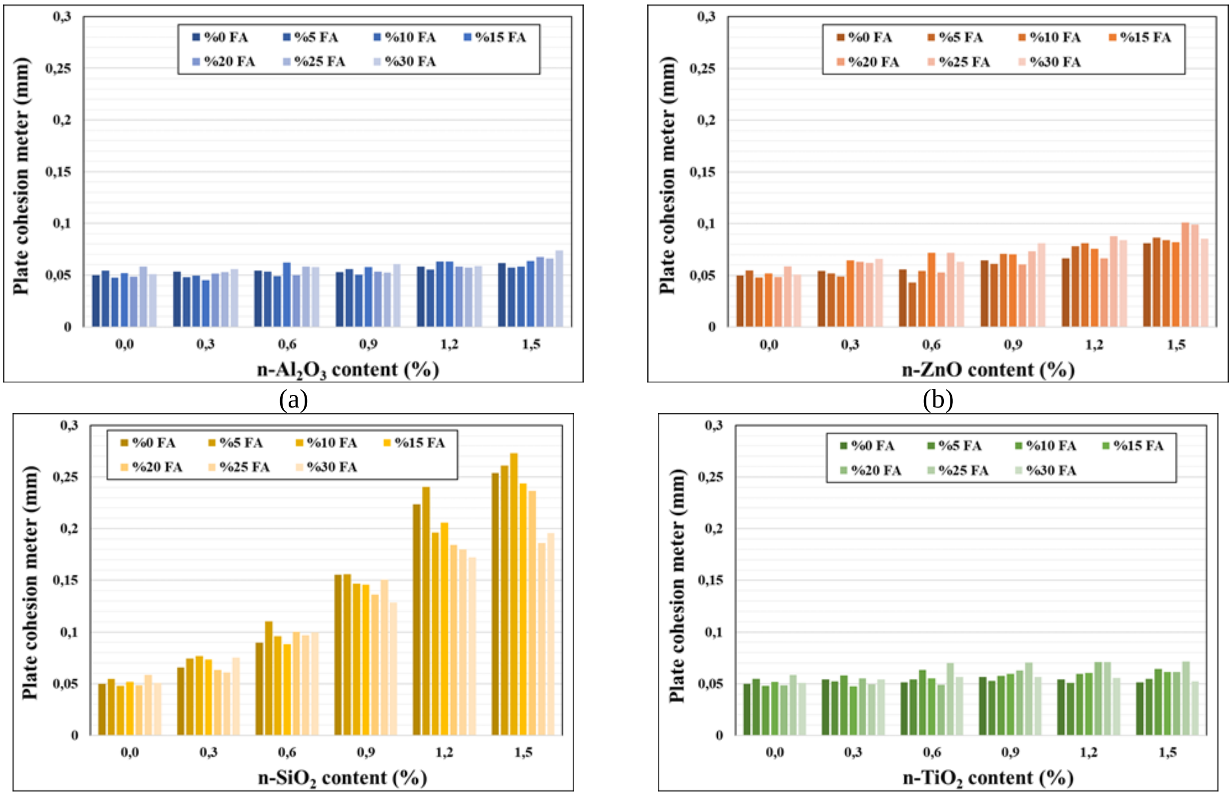
Fig. 8 Mini slump flow diameters of the samples with respect to change of CNPPs amount



(a) (b)

(c) (d)

Fig. 9 Marsh cone flow time of the samples with respect to change of CNPPs amount



(a) (b)

(c) (d)

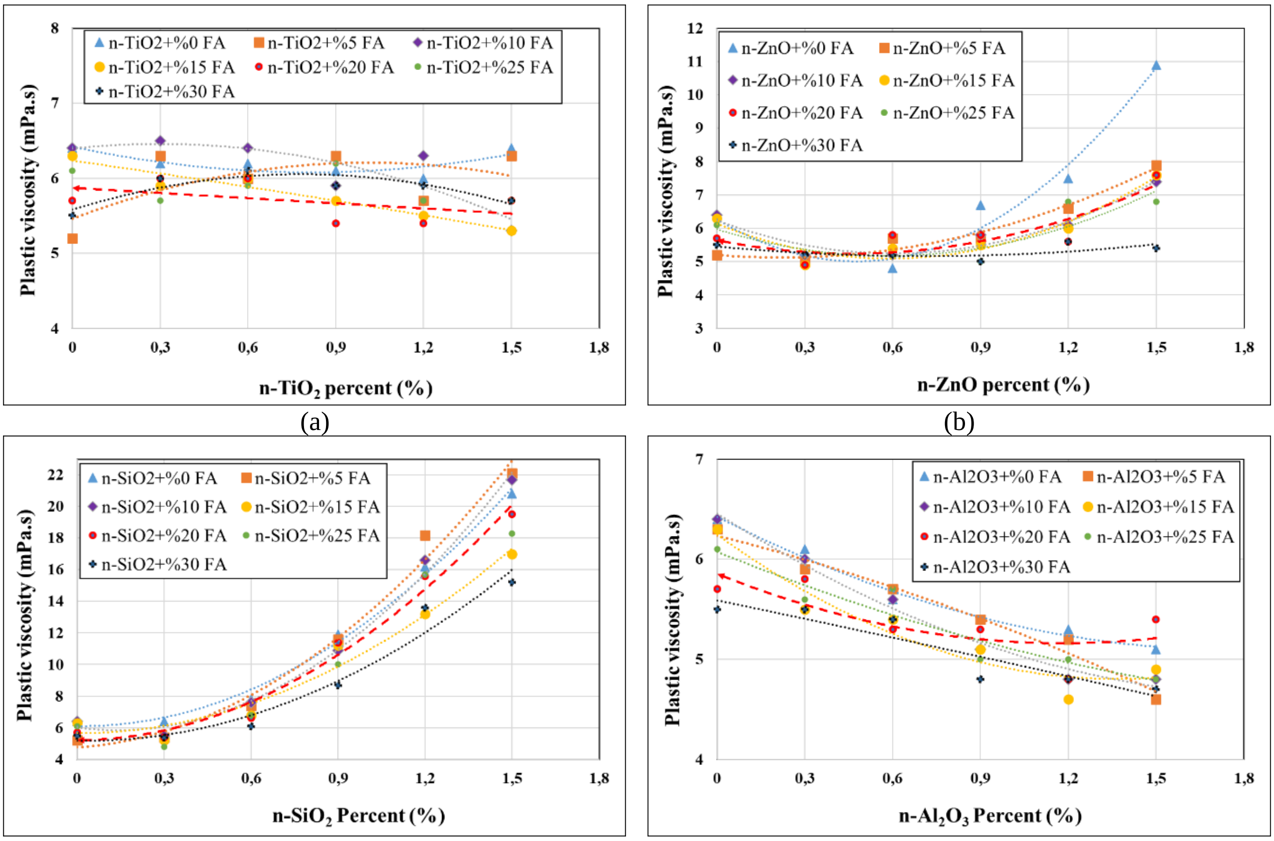
Fig. 10 Plate cohesion meter values of the samples with respect to change of CNPPs amount

content of nanomaterials can be explained by the specific surface areas of these nanomaterials.

The increase in n-SiO_2 content in the grout mixes resulted in an increase in the marsh cone flow time of the grout mixes more than the increase caused by these three nanomaterials (Fig. 9). This increase can be thought as remarkable for the samples prepared with addition of n-SiO_2. This increase can be explained as an increase in the cohesion value of the grout mixes due to the very low specific surface area of n-SiO_2. It can be also explained as all CNPPs have different densities from each other as shown in Table 3. According to this table, n-TiO_2, n-ZnO, n-Al_2O_3 and n-SiO_2 have densities 4100, 5500, 3900 and 2200 kg/m^3, respectively. As can be clearly seen from these values, n-SiO_2 has the lowest density while n-ZnO has the highest density. Since CNPPs are added by weight to the mixtures in the preparation of the grout samples, the volumetric effect of each CNPPs in the mixture will be different due to the differences in their densities. Due to the fact that n-SiO_2, which has the lowest density among all nano materials, has a higher volume in the mixtures, it is thought to cause much more significant changes on the fluidity behavior of mixtures compared to other nanomaterials used in this study. n-SiO_2 has noticeable effect on the arranging of required water amount for cement-based grouts (Liu *et al*. 2012). For this reason, an increase is observed in the resistance of grout mixtures against flowing. Furthermore, as can be clearly seen from Fig. 8 and Fig. 9, it was determined that the mini slump spreading diameter and marsh cone flow time behave in harmony with each other with the addition of CNPPs of the FA added grout mixtures.

As seen in Fig. 10, although the increase in the ratio of n-TiO_2 and n-Al_2O_3 addition to the grout mixtures caused a small increase in the plate cohesion values of the samples, this increase cannot be considered as very remarkable increase on the fluidity features of these samples. Furthermore, plate cohesion values of n-ZnO added grout mixtures increased more compared to these two nano materials with the increase of nano material content (see Fig. 10). The highest rate of increase in the plate cohesion values of the grout mixtures was observed in the n-SiO_2 added grout mixtures as seen in Fig. 10.

This increase is evaluated as a dramatic increase. This dramatic increase can be explained as an increase in the cohesion value of the grout mixes as same as marsh cone flow time and mini slump diameter values of the grout samples due to the very low specific surface area and very low density of n-SiO_2 when comparing with the other CNPPs used in this work (Zabihi and Ozkul 2018, Balapour *et al*. 2018, Ouyang *et al*. 2018, Oltulu and Sahin 2013). This similar behavior was also observed in the past studies related with using of CNPPs as additive in grout mixtures (Peng *et al*. 2019, Rashad 2013, Singh *et al*. 2012, Senff *et al*. 2009). In addition, as it is clearly seen from the results FA substitution in the grout mixtures causes decrease of plate cohesion values of the samples. The cohesion-increasing effect of CNPPs is limited by adding FA (Zabihi and Ozkul 2018, Balapour *et al*. 2018, Ouyang *et al*. 2018, Oltulu and Sahin 2013). This limitation can be considered as an improvement in the workability behavior of the respective grout mixes.



(a) (b)

(c) (d)

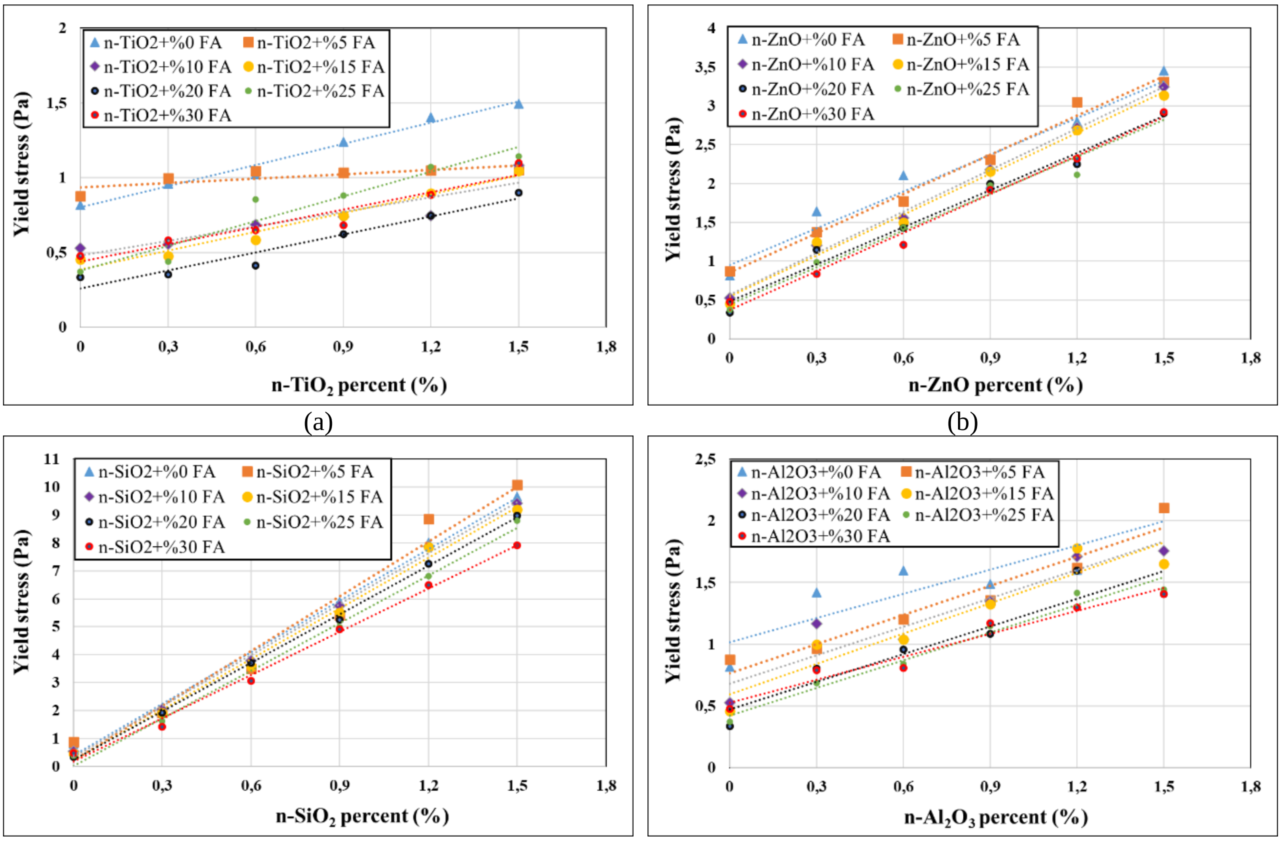
Fig. 11 Plastic viscosity changes with different addition ratio of CNPPs and FA amounts

3.2 Rheological test results of the samples

The effects of four different types of Colloidal Nano Particular Powders (CNPPs) ($n-SiO_2$, $n-TiO_2$, n-ZnO and $n-Al_2O_3$) additions at different amounts by mass (%0.0, %0.3, %0.6, %0.9, %1.2 and %1.5) on the rheological properties of cement-based grouts incorporated with fly ash as mineral additive at different constitutes (%0-for control purpose, %5, %10, %15, %20, %25 and %30) were investigated and discussed in this part.

As seen in Fig. 7, shear stress-shear rate graphs were drawn for all grout mixtures, and rheological parameters (such as plastic viscosity and yield stress) were obtained from these graphs by considering the Modified Bingham model. Shear thickening behavior was observed for all grout samples when the graphics were examined. The dilatant behavior was determined from the curves drawn to evaluate the rheological parameters of all grout mixtures as seen in Fig. 7. This expected behavior has also been observed in some previous studies (Celik and Canakci 2015, Celik and Akcuru 2020, Cry *et al*. 2000, Yahia and Khayat 2001). This flow behavior can be described by the regular-irregular transition theory proposed by Hoffman (1998). This theory is based on flow behavior that changes from a regular to an irregular state at a given critical shear rate. Most of the flow energy is absorbed by collisions between particles during agglomeration in less ordered structures to obtain suspension flow (Celik and Canakci 2015). Therefore, as the shear rate in the flow increased, the apparent viscosity and shear stress increased exponentially in all mixtures prepared for this study due to dilatant behavior. This means that higher energy is needed at higher shear rates for such grout mixes that show dilatant behavior. Furthermore, the binders (cement and FA for this study) are subjected to a partial flocculation due to the increased water content of the grout mixes at high w/b ratios (w/b > 1.0). This partial flocculation can be described by the hydrodynamic interaction between particles observed at higher shear rates (Celik and Canakci 2015). As a result, the clumping caused by partial flocculation may require greater pressure on the grout suspension and reduce the fluidity of the grout mixes. Therefore, the suspension particles in grout matrixes during flow may need more energy at higher shear rates, resulting in an increase in apparent viscosity values (Wagner and Brady 2009). Therefore, observing the effect of nano-sized additives in such cement-based grout mixtures with high fluidity can present remarkable effects.

Plastic viscosity and yield stress changes with different addition ratio of CNPPs and FA amounts are shown in Figs. 11 and 12. As can be clearly seen from Fig. 11, $n-TiO_2$ mixed grout mixtures prepared by adding FA have completely different plastic viscosity change from ones prepared without adding FA. While plastic viscosity decreases up to a certain amount of $n-TiO_2$ (0.6%) for the grout samples prepared in different $n-TiO_2$ substitutions without FA additives, it starts to increase again after this ratio is exceeded (see Fig. 11). The smallest plastic viscosity was obtained at a rate of approximately 0.6% n-



(a) (b)

(c) (d)

Fig. 12 Yield stress changes with different addition ratio of CNPPs and FA amounts

TiO_2 proportion. This proportion is seen as the place where the fluidity is highest. The behavior of grout samples prepared with FA and n-TiO_2 additives shows different changes in plastic viscosity values. While plastic viscosity increases up to a certain n-TiO_2 amount with the increase in n-TiO_2 amount in all FA additive ratios, when these values are exceeded, the plastic viscosity values decrease again, which means that the fluidity increases. Rising amount of n-TiO_2 in grout mixtures has made the plastic viscosity of the samples with FA considerably decrease (see Fig. 11). The addition of n-TiO_2 has made the flowability of cement-based grouts containing FA remarkably increase. According to Fig. 11, the highest reduction for the plastic viscosity in the grout samples with n-TiO_2 addition has been observed in the mixtures with 15% fly ash. After this ratio, the plastic viscosity values of the n-TiO_2 additive grouts again have started to increase.

Yield stress changes based on addition of FA to the grout samples has been considerably influenced as shown in Fig. 12. According to Fig. 12, the increase amount of FA content in all grout mixtures with different proportions of n-TiO_2 has made the yield stress of fresh grout mixtures increase. The lowest yield stress values were measured in the grout samples prepared by addition of 20% FA for all n-TiO_2 contents. All results show that the yield stress value of the samples have been improved because of the usage of FA as a mineral additive in cement-based grouts. Since n-TiO_2 has very low specific surface area, addition of this CNPP to the grout samples has caused an increase on the yield stress values of the samples. Because of having such a low specific surface area, the water requirement of the grout matrix tends to increase. As a result of this, cohesion or yield stress of the grout samples has started to increase.

As shown in Fig. 11, for all grout mixtures with n-ZnO additives, the increase in n-ZnO amount decreases certain amount of plastic viscosity values up to 0.6% additive rate. At this proportion, all plastic viscosity values of the grout samples prepared with or without FA meet at the nearly same value. After this proportion is exceeded, plastic viscosity values of the samples again have started to increase. While the highest increase in the plastic viscosities was observed in grout mixes without FA additives, the lowest increase was observed in grout mixtures with 30% FA added (see Fig. 11). While the plastic viscosity value of the grout sample prepared with 30% FA additive was measured as 5.4 mPa.s with 1.5% n-ZnO additive, the plastic viscosity value of the FA additive-free grout sample was measured as 11 mPa.s with 1.5% n-ZnO additive (see Fig. 11). Therefore, this shows that the use of n-ZnO as an additive together with FA in grout mixtures will result in a significant decrease in plastic viscosity values. The yield stress changes of the grout samples prepared with addition of FA and n-ZnO are shown in Fig. 12. As can be clearly seen from Fig. 12, the yield stress values increased linearly with the increase of n-ZnO additive ratio in all grout mixtures. The meaning of the increase on the yield stress of the samples can be explained as increase of cohesion in the sample (Zabihi and Ozkul 2018, Balapour *et al.* 2018). As can be seen in Table 3, the specific surface areas determined for n-ZnO was measured as 70 m^2/g. This small surface area increases the amount of water required to cover the surface of n-ZnO, and this affects the amount of water needed by the mixture. The use of n-ZnO and FA together in grout mixes slightly affects the yield stress value of grout mixes. According to Fig. 12, the increase in the FA amount in all n-ZnO additives causes the yield stress values of the mixtures to decrease. This shows that the use of n-ZnO and FA together in grout mixtures can lead to an improvement in the yield stress values of the mixtures.

The plastic viscosity changes values of n-SiO_2 and FA added grout mixtures are shown in Fig. 11. As shown in Fig. 11, plastic viscosity values of all grout mixtures exponentially increase with respect to increase amount of n-SiO_2. This increase can be explained as the water requirement of the cement-based matrix shows an increase with rising of nano silica content because of having large specific surface area (Zabihi and Ozkul 2018, Balapour *et al.* 2018). According to Table 3, the specific surface area of n-SiO_2 was measured as 650 m^2/g. This value is so higher than the other CNPPs used in this study. This CNPP has also very low density (2200 kg/m^3) when comparing with the other CNPPs used in this work (Zabihi and Ozkul 2018, Balapour *et al.* 2018, Ouyang *et al.* 2018, Oltulu and Sahin 2013). Since n-SiO_2, which has the lowest density among all CNPPs, has a higher volume in the grout mixtures, it causes much more remarkable changes on the fluidity behavior of grout mixtures compared to other CNPPs used in this study. This similar behavior was also observed in the past studies (Peng *et al.* 2019, Rashad 2013, Singh *et al.* 2012, Senff *et al.* 2009). n-SiO_2 has considerable influence on the arranging of required water amount for cement-based grouts (Liu *et al.* 2012). Therefore, exponential increase is observed in the resistance of grout mixtures against flowing. In addition, as it is clearly seen from the results FA substitution in the grout mixtures causes decrease of plastic viscosity values of the samples. The fluidity-increasing effect of n-SiO_2 is limited by adding FA (Zabihi and Ozkul 2018, Balapour *et al.* 2018, Ouyang *et al.* 2018, Oltulu and Sahin 2013). This limitation can be considered as an improvement in the fluidity behavior of the respective grout mixes prepared with addition of n-SiO_2. The lowest plastic viscosity values in all grout mixtures were measured in 30% FA added grout samples for all n-SiO_2 substitutions (see Fig. 11). The yield stress changes of the grout samples prepared with addition of FA and n-SiO_2 are also seen in Fig. 12. According to this figure, the increase of n-SiO_2 additive ratio in all grout mixtures has made the yield stress of grout mixes incrementally increase. This increase can be defined by increase of cohesion in the sample (Zabihi and Ozkul 2018, Balapour *et al.* 2018). This cohesion is directly related with the specific surface area and density of n-SiO_2 as same as the other CNPPs. Because of having such a very low specific surface area and density comparing with the other CNPPs, n-SiO_2 causes the need for large amounts of water in the grout mixtures. Therefore, this situation makes the yield stress of the samples increase with respect to increase amount of n-SiO_2. Moreover, the use of n-SiO_2 and FA together in grout mixes slightly decreases the yield stress value of grout mixes. This shows that the use of n-SiO_2 and FA together in grout mixtures can lead to an improvement in the yield stress values of the mixtures. The lowest yield stress values in all grout mixtures were measured in 30% FA added grout samples for all n-SiO_2 substitutions (see Fig. 12).

The effects of usage of n-Al_2O_3 and FA additives in the grout mixtures on plastic viscosity values are shown in Fig. 11. Plastic viscosity values of all grout mixtures dramatically decrease with respect to increase amount of n-Al_2O_3 different from the other CNPPs used in this study (see Fig. 11). Furthermore, increase amount of n-Al_2O_3 in grout mixtures has made the plastic viscosity of the samples with FA slightly decrease (see Fig. 11). The addition of n-Al_2O_3 has made the flowability of cement-based grouts containing FA remarkably increase. The reduction of the plastic viscosity with increase amount of n-Al_2O_3 can be explained by higher ettringite contents that can be observed by the initial dissolution of nano alumina at the early hydration process (Oltulu and Sahin 2013, Liu *et al.* 2012). Since it directly participates in the hydration process, it does not have a negative effect on the fluidity of the grout mixture. Moreover, it also contributes to the increase of the fluidity of the grout mixture. The yield stress changes of the grout samples prepared with addition of FA and n-Al_2O_3 are shown in Fig. 12. These changes are very similar to the changes observed for n-ZnO added grout mixtures mentioned above. the yield stress values increased linearly with the increase of n-Al_2O_3 additive ratio in all grout mixtures. The increase of cohesion in the samples with addition of n-Al_2O_3 causes an increase in the yield stress of the grout samples (Zabihi and Ozkul 2018, Balapour *et al.* 2018). This increase is directly related with the specific surface area (140 m^2/g) of n-Al_2O_3. The addition of n-Al_2O_3 and FA together in grout mixes slightly decreases the yield stress value of grout mixes. This indicates that the addition of n-Al_2O_3 and FA together in grout mixtures can lead to an improvement in the yield stress values of the grout mixtures.

3.3 Comparison results of CNPPs based on rheological properties

As can be clearly seen in Fig. 13, the plastic viscosity values of cement-based grouts are given comparatively, depending on different FA mineral additives in 4 different CNPPs additive ratios. As can be understood from the figure, plastic viscosity values for each CNPP show serious differences depending on different FA additives.

In all FA and n-SiO_2 additive blends, plastic viscosity values showed significant exponential increases due to the increase in the nano powder ratio compared to other CNPPs (see Fig. 13). This means that n-SiO_2 increases plastic viscosity in grout mixtures independent of FA additive and therefore reduces fluidity. This increase in plastic viscosity based on increase amount of n-SiO_2 is quite large compared to other CNPPs. From here, adding more than 0.3% of n-SiO_2 with or without FA additives to cement-based grouts does not contribute much in terms of fluidity behavior. For addition of n-ZnO, the plastic viscosity changes are slightly

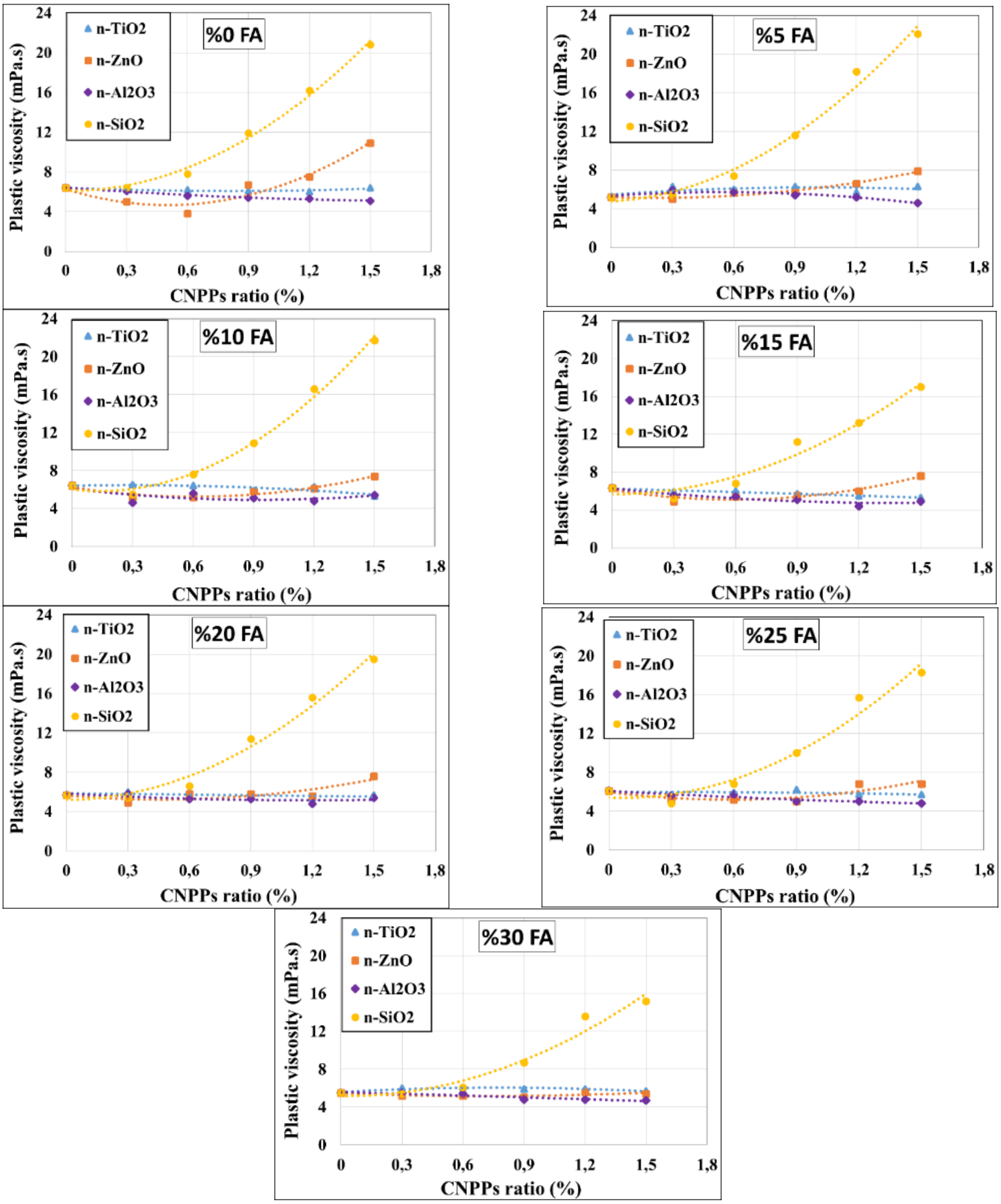


Fig. 13 Effect of different CNPPs usage on the plastic viscosities based on incorporations of different substitution rate for FA

different from other CNPPs. As can be clearly seen from Fig. 13, the increase in the amount of n-ZnO up to 0.6% in FA additive-free grout mixtures reduced the plastic viscosity values to the lowest values compared to all other CNPPs used in this study. After this ratio, plastic viscosity values started to increase with addition of n-ZnO and higher plastic viscosity values were measured in grout mixtures after 0.9% proportion compared to n-Al_2O_3 and n-TiO_2 additives (see Fig. 13). However, the increase in the FA substitution as a mineral additive has started to reduce this parameter again in the part of the n-ZnO additive that increases the plastic viscosity. Then, it has enabled n-Al_2O_3 and n-TiO_2 additives to take values close to plastic viscosity values. As can be seen in Fig. 13, the effects of n-Al_2O_3 and n-TiO_2 on grout mixtures have been observed as similar behavior in cases with and without FA additives. Plastic viscosity values due to the increase in the addition rates of both nanomaterials did not show a very noticeable tendency to decrease or increase. They tended to decrease only slightly in each amount. In addition, the inclusion of these

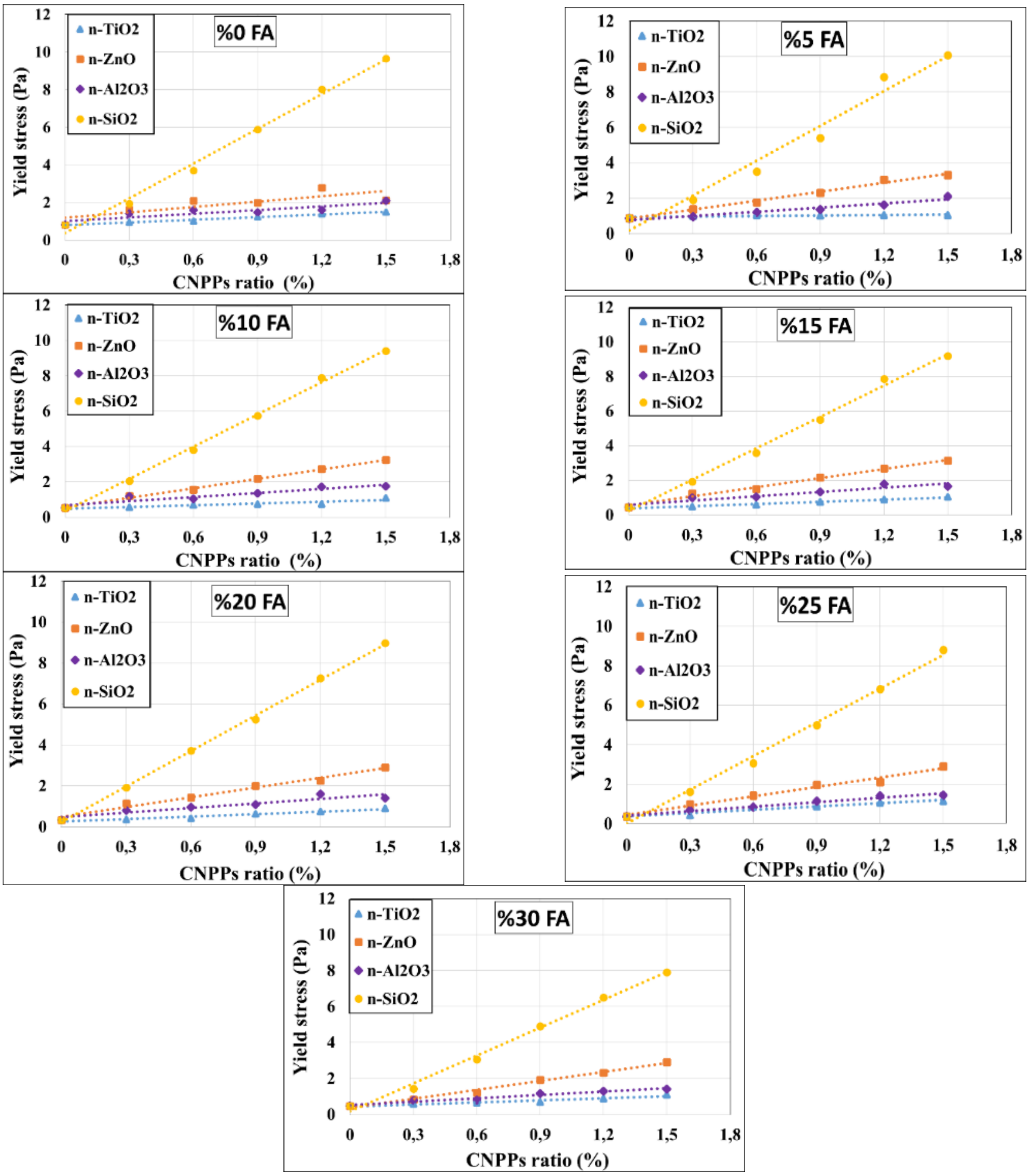
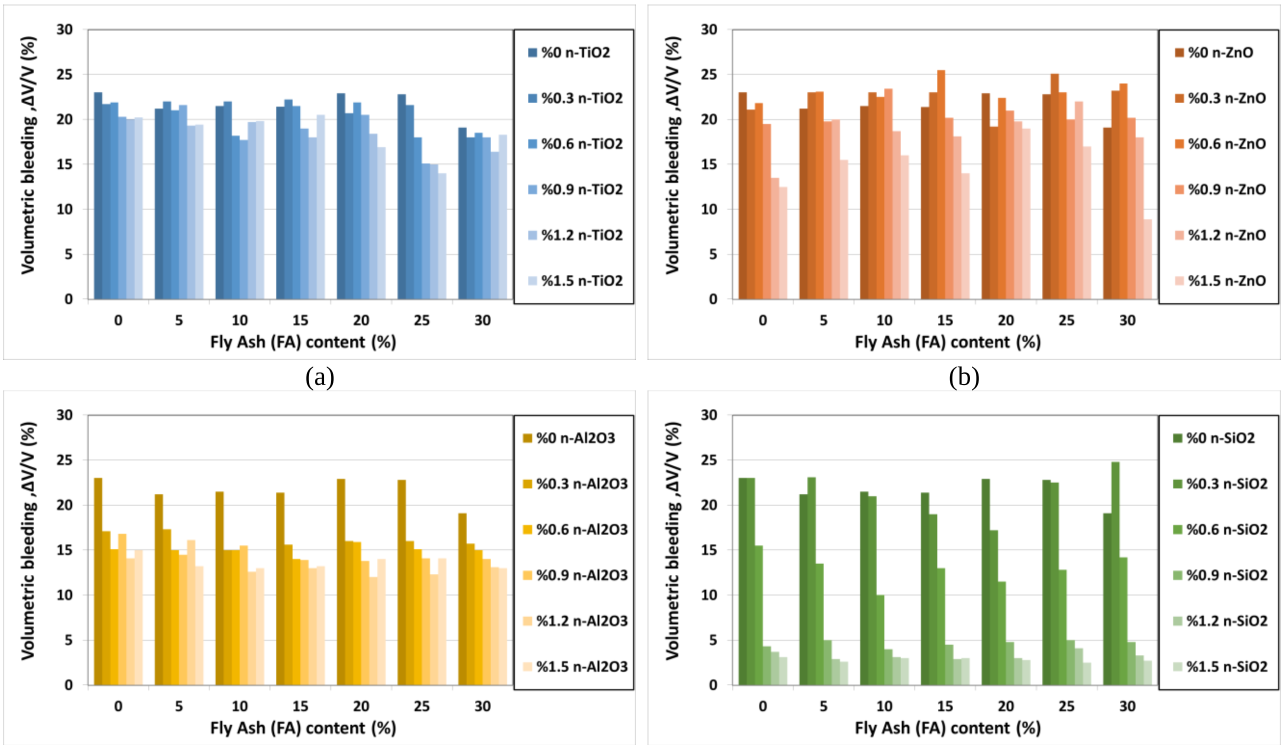


Fig. 14 Effect of different CNPPs usage on the yield stresses based on incorporations of different substitution rate for FA

nanomaterials in grout mixtures with FA did not change the results so much comparing to the other CNPPs (such as n-SiO_2 and n-ZnO). Nano alumina tended to decrease depending on the rate of increase in nanomaterial amount in the mixtures, albeit a small amount compared to nano titanium oxide. Nevertheless, the lowest plastic viscosity value among FA mineral additive mixtures was reached at the rate of 5% FA additive and 1.5% n-Al_2O_3 additive.

As can be clearly seen in Fig. 14, the yield stress values of cement-based grouts are given comparatively, depending on different FA mineral additives in different additive ratios of four different nano materials. As can be understood from the Fig. 14, plastic viscosity values for each nanomaterial show some changes depending on different FA additives. The increase in the rate of nano material in all mixtures with and without FA additives caused a linear increase in yield stresses. Among all CNPPs, the highest yield stress values measured due to the increase in the nanomaterial ratio were observed in n-SiO_2 doped mixtures. Yield stress increases linearly with a high slope when n-SiO_2 content increases in grout mixes with or without FA additives. As discussed in the previous paragraph, the highest plastic viscosity value was determined in grout mixtures with n-SiO_2 additives. This situation in yield stress is also parallel to the plastic viscosity behavior. The main reason of this increase has been discussed detailly in previous paragraphs.



(a) (b)

(c) (d)

Fig. 15 Volumetric bleeding ratios of all mixtures prepared by addition of CNPPs and FA

Contrary to this increase, the lowest yield stresses were detected in n-TiO_2 added mixtures. Although the increase in the amount of FA in grout mixtures decreased the yield stress values of all nano-added grouts (especially in n-SiO_2 added mixtures), this reduction cannot be considered as remarkable decrease. The greatest effect was observed on mixtures with n-SiO_2 additives. It is thought that this situation can be explained by the fact that the particle sizes of the FA used within the scope of the study are close to the particle sizes of the cement. The increase in yield stress is expressed as the increase in the resistance to flowing of cement-based grouts, especially in the situation just before they start to flow. For this reason, it is not desirable to increase this value in mixtures with high fluidity.

3.4 Bleeding test results of the samples

Bleeding tests were carried out on the grout mixtures prepared within the scope of this experimental study. As a result of the experiments, bleeding values expressing the stability of the mixtures were determined. Volumetric bleeding ratios of all mixtures are shown in Fig. 15. As can be seen in Fig. 15, the bleeding values of all mixtures with and without n-TiO_2, n-ZnO and n-Al_2O_3 remained below 900 ml (above 10% volumetric bleeding rate). Only 30% FA mineral added 1.5% n-ZnO remained above this rate in the mixtures. However, the bleeding rates obtained in mixtures with n-Al_2O_3 additives have given lower results than the bleeding rates obtained in both n-TiO_2 and n-ZnO. n-Al_2O_3 was more effective than the other two nano additives in terms of contribution to stability. As can be seen in Fig. 15, the bleeding values of the grout mixes with and without mineral additives of 0.9% or more with n-SiO_2 additives remained above 900 ml (below 10% bleeding rate). This behavior is different from the behavior of all nano-additives mentioned before. FA substitution as a mineral additive to the grout mixes with n-SiO_2 does not have a significant effect on bleeding results as with other nanomaterials. On the contrary, a certain amount of decrease is observed in the bleeding values due to the increase of n-SiO_2 in all FA additive and additive-free blends (see Fig. 15). It can be said that the addition of n-SiO_2 improves the bleeding values of mortar mixtures, even if it is small, like other nanomaterials. In addition to all these, n-SiO_2, which will be used as an additive at a rate of 0.9% or more, has reduced the bleeding rate of the grout mixtures below 10%, thus contributing to the stabilization of these mixtures.

Bleeding rate above 10% is not a highly desirable situation in terms of the stability of the grouts (Deere 1982). As can be seen in the rheological and fluidity parameters of these mixtures, the fluidity of the mixtures was seriously affected by these bleeding behaviors. Especially the high bleeding values caused the development of shear thickening behavior in the shear rate-shear stress graphs, which is one of the rheological parameters of mortar mixtures (see Fig. 7). It has emerged as the development of high shear stresses at high shear rates (this situation represents high injection pressure in soil injection applications) and consequently high apparent viscosity values. The reason why the bleeding values are so high can be explained by using the water-binding ratio (w/b=1), which is considered high. This water-binder ratio is a value generally used in the permeation and jet grouting methods in ground improvement applications. The main purpose of selection this water to binder ratio in this study was considered as investigating these types of ground improvement applications.

4. Conclusions

The conclusions that can be drawn from the study are shown as given below.

- Although CNPPs are in the same amount in the mixtures, the reason why these CNPPs have different effects on workability, rheological and stability features of the cement-based grouts is that these nano-sized materials have different grain sizes, surface areas and densities.
- The increase in the specific surface area due to the nano size increased the water retention capacity of the mixtures, which somewhat limited the fluidity behavior of the grout mixtures.
- Since the CNPPs examined in this study had different densities, the volumetric effects of these CNPPs in the grout mixtures were different, although they were added at a constant mass amount. This caused the fluidity and stability behaviors of the mixtures to differ in different CNPPs additives.
- Workability and rheological test results showed that the usage of CNPPs in the grout samples caused to increase in cohesion values (yield stress) of the grout samples because of very large specific surface area.
- Shear thickening behavior was observed for all grout samples according to the rheological test. The dilatant behavior was determined for all grout mixtures. This means that higher energy is needed at higher shear rates for such grout mixes that show dilatant behavior.
- Although the use of CNPPs as an additive material in grout mixtures at high w/b ratios (w/b>1.00) limited the fluidity somewhat, it has contributed to the disappearance of shear thickening behavior.
- All bleeding test results showed that the addition of CNPPs improved the bleeding values of the grout mixtures. The high bleeding values caused the development of shear thickening behavior. Therefore, this reduction in the bleeding values is considered as an improvement for stability features of the grout samples.

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