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Rheological behavior of mixtures used in 3d-printing: experimental evaluation of the effectiveness of criteria requirements for filler

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ABSTRACT

Introduction. For handling the problem of mixtures design for additive construction technologies, the paper presents the results of experimental studies of rheological behavior and production characteristics (plasticity and shape stability) of cement mixtures based on various types of fillers with different size, shape, and grade. **Methods and materials.** Rheological properties of 3D-printable mixtures were investigated using squeezing rheometry methods. The constant strain rate mode of 5 mm/s was used to evaluate plasticity and the constant load rate of 5 N/s was used to evaluate form stability. Scanning electron microscopy method (Phenom XL) was used to evaluate the size-geometry characteristics of cement and filler particles. Image processing to determine particle length and width was performed using ParticleMetric software. The size and gradation of the cement and filler particles were evaluated using a laser particle size analyser "Analyzette 22". **Results and discussion.** It was found that a necessary condition for the plasticity and stability of mixtures is the creation of dense spatial packing of disperse phase particles. The values of the plasticity limit rational for extrusion are ensured if the filler particles have a size comparable to cement particles and multi-size gradation. The characteristics of the fillers are not decisive for the shape stability of the mixtures. **Conclusion.** The numerical criteria of fillers for design of 3D-printable mixtures have been substantiated, including mean average particle diameter, particle shape factor, particle distribution constant as a characteristic of the particle size gradation.

KEYWORDS: additive technologies, cement mixture, fillers, rheology, workability

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INTRODUCTION

The development of 3D-build printing creates the need for a wide range of mixtures on the market that meet the requirements of this technology. The efficiency of 3D-build printing is determined by a number of critical properties of mixtures such as plasticity for implementation of extrusion (extrudability), shape stability in multilayer casting (buildability), structural build up [1-4]. As a result of the accumulation of a number of experimental data, e.g. [5-19], a lot of different types of mixtures with the necessary technological properties for printing have been developed and successfully tested. The obtained mixtures are characterised by their multi-component mix design, their compositions simultaneously using superplasticisers, viscosity modifiers, hydration process regulators, fillers and aggregates of different chemical and mineralogical composition and particle size.



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When designing mixtures, the role of binders, plasticisers and viscosity modifiers in regulating technological properties is theoretically justified and established experimentally [6–8, 10–11]. At the same time, the choice of fillers and the regulation of their concentration in the mixture compositions is based on an empirical approach [12–18]. When developing compositions, the filler content of a certain type is selected without defining the general requirements for their properties, which are necessary for a priori (before experience) determination of the applicability of a particular filler in 3D build printing and for determining the limits of its rational content.

The author's approach to the modelling and control of the rheological behavior of visco-plastic mixtures in 3D printing processes and the optimization of their mix design is based on the provisions of the structural rheology of disperse systems, the priority of whose theoretical foundation belongs to the Soviet school of physico-chemical mechanics [19–20]. On this basis, the main means of controlling the rheological behavior of viscoplastic mixtures considered as heterogeneous systems "disperse phase + dispersion medium" have been substantiated by author team [21]. The criteria requirements for the properties of fillers are theoretically justified and numerical criteria for their evaluation are proposed [22]. These criteria include average particle diameter d, particle shape factor k_s, particle distribution constant G_{nc}, which are proposed to be used for preliminary complex evaluation of fillers in the problems of mixture design.

This paper discusses the results of experimental evaluation of the effect of numerical filler criteria on the rheological behavior and properties of 3D printable mixtures.

MATERIALS AND METHODS

Five types of mixtures were investigated (Table 1). The parameters of mix composition such as cement: filler mass ratio, concentration and type of additives, fiber, W/C-ratio were kept constant because this parameters were established according to the results of the previous studies [23–24]. The variable factor in the mix composi-

Table 1Mix composition

tion was the size-geometry characteristics of the fillers. The mixtures on 4 types of fillers were studied such as aleuropelite (Al), fly ash (FA), crushed sand (CS), crushed limestone (CL). The characteristics of the mixture initial components are presented in Table 2.

Methods for evaluating filler properties. Scanning electron microscopy (SEM) was used to evaluate the shape of the particles. Imaging was performed on a Phenom XL scanning electron microscope ($v_{acc} = 15 \text{ kV}$. P = 0.10 Pa). The images were processed using the ParticleMetric software in order to determine particle length *l* and width *b*. The average particle diameter and granulometric constant were calculated based on the data obtained on a laser particle size analyzer Analysette 22 Nano Tec.

The numerical filler criteria were calculated from the data obtained:

1) average particle diameter d_c

$$d_c = \frac{c_1 d_1 + c_2 d_2 + c_3 d_3 + \dots + c_{i+1} d_{i+1}}{c_1 + c_2 + c_3 + \dots + c_{i+1}}.$$
 (1)

where c_i is partial residuals on sieves. %; d_1 is diameter taken as average for a certain interval (fraction). μ m;

2) particle shape factor k_s , calculated as the arithmetic average of two linear sizes (length *l*. width *b*):

$$k_s = (l+b)/2.$$
 (2)

3) particle distribution constant G_{pc} as characteristic of the particle size gradation:

$$G_{pc} = d_{60}/d_{10},\tag{3}$$

where $d_{10}(d_{60})$ are diameters of particles. less than which the material contains 10% (60%) particles by weight. respectively.

Test parameters for evaluating the mixture rheological properties. The method of squeezing rheometry was used. The squeezing test were carried out on cylindrical samples of the fresh mixtures, the radius of which R, was equal to their height, $h_0 = 25$ mm. The specimens were moulded and tested immediately after the preparation of

System ID	Viscosity modifier dosage, % mass cement		Superplasticizer dosage, % mass	Cement : filler	Fiber dosage, %
	XG	SG	cement	massiano	mass cement
C (reference)	0.2	0.2	0.2	1:1	0.3
C+Al	0.2	0.2			
C+FA	0.2	0.2			
C+CS	0.2	0.2			
C+CL	0.2	0.2			

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Table 2Materials characteristics

Material type	Brand	Characteristics
Cement (C)	CEM I 42.5R, "Sukholozhskcement branch of LLC SLK Cement"	$C_3S - 61.7\%$. $C_2S - 14.9\%$. $C_3A - 5.6\%$. $C_4AF - 12.6\%$
Superplasticizer (SP)	Sika®Visco Crete [®] 20HE	Polycarboxylate ethers
Xanthan gum (XG)	FUFENG [®] 80. "Xinjiang Fufeng Biotechnologies Co", China.	$(C_{35}H_{49}O_{29})_n \sim 91\%$
Silicate glass (SG)	Liquid glass technical (according to GOST 13078-81), "NPO Silikat"	SiO ₂ – 33.76%. Na ₂ O – 66.24%
Aleuropelite (Al)	Bigila deposit of the Ishim Formation, Tyumen Region	$SiO_2 - 81\%$. K[AlSi ₃ O ₈] - 3%. Na[AlSi ₃ O ₈] - 10%. PbSO ₄ - 3.1%
Fly ash (FA)	Reftinskaya HPP. Reftinsky settlement. Berezovsky town. Sverdlovsk region	$ \begin{array}{l} {\rm SiO_2-60.0\div 62.0\%.\ Al_2O_3-29.0\div 31.0\%.} \\ {\rm Fe_2O_3-4.0\div 5.0\%.\ CaO+MgO-1.5\div 2.5\%} \end{array} $
Crushed sand (CS)	"Khrustalnaya Gora Khrustalnaya Quarry", Sverdlovsk region	SiO ₂ ~ 98%
Crushed limestone (CL)	«MP-2». "Polevskoy Marble"	CaCO ₃ ~95%
Fiber (F)	"C-Airlaid", Chelyabinsk	Polypropylene 100%, 12 mm in length, $d = 20-25 \mu m$.

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the mixtures. For squeezing test, the specimen was placed between two smooth plates with a diameter corresponding to the specimen size and loaded into an INSTRON 3382 floor hydraulic testing system. Two loading modes were applied during the tests:

1) with a constant specimen deformation rate of 0.5 mm/s, modelling the visco-plastic flow of the mixture under dynamic conditions during extrusion (method of N. Roussel [26]);

2) with a constant load rate of 5 N/s, simulating the behavior of the mixure under static conditions of layerby-layer casting (author's method). [22]).

As a result of squeezing tests, the rheological behavior of the mixtures was evaluated by analysis of experimental curves:

- "load N relative change in specimen height h_i/R" obtained from constant specimen deformation rate tests;
- "load P displacement Δ " obtained from constant load rate tests.

The curves "N $-h_i/R$ " were obtained during the experiments were interpreted as influence curves of reduced compression load F* from a relative change of height of the sample h_i/R : ("F* $-h_i/R$ "):

$$F_i^* = Nh_i / \pi R^2, \tag{1}$$

where $h_i = (h_0 - \Delta)$, h_0 is the initial height of the sample, Δ is transferred in the point of time, value R was taken as constant and equal to the radius of the sample at the beginning of the experiment.

The values of structural (σ_0) and plastic strength (σ_{pl}) of mixtures at the moments corresponding to the beginning of deformation ($\Delta = 0.1$ mm) and the beginning of cracking of specimens were calculated from the obtained experimental "P- Δ " curves according to the formula:

$$\sigma = P/\pi R^2.$$
 (2)

The following properties of the mixtures were determined to be suitable for 3D printing based on the test results:

- yield stress value $K_i(I)$ was calculated at the inflection point of the "F* - h_i/R " curves $(h_i/R = 0.9)$:

$$K_i(h/R) = \frac{\sqrt{3}F^*}{2}; \qquad (3)$$

- structural strength (σ_0) as the ability of the mixture to withstand the load without deformation;
- plastic strength (σ_{pl}) and relative plastic strains $(\Delta_{pl} = \Delta/h_0)$ characterising the ability of the mixture to withstand load without cracking.

RESULTS

Size-geometry characteristics of the fillers. All types of fillers used are characterised by continuous gradation (Table 3, Fig. 1), which, as previously demonstrated [10, 27], is a necessary condition for ensuring the workability of 3D-printable mixtures according to the criteria of plasticity and shape stability. The particle distribution constant of the fillers is in the range of $G_{pc} = 7.6-9.9$.



Table 3Particle size distribution

	ω of particles, %				
Particle size d, m	Aleuropelite	Fly ash	Crushed sand	Cement	Crushed limestone
≤ 4	16.8	9.1	11.3	11.8	11.5
8	14.6	8.2	6.3	9.0	6.8
15	17.5	14.6	10.2	13.9	15.2
30	28.7	28.1	29.0	27.5	27.4
55	20.1	29.0	33.6	28.2	16.8
100	2.3	10.9	9.6	9.6	9.8

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Fig. 1. SEM-images of filler particles: a) Aleuropelite; b) Fly ash; c) Crushed sand; d) Crushed limestone

Its value for Portland cement is in the same range ($G_{pc} = 8.1$) (Table 4).

The fillers differ significantly in shape and size. Flat particles of aleuropelite (Fig. 1a) are characterised by

the shape factor $k_s = 2.33$. Spherical particles of fly ash (Fig. 1b) are characterised by the shape factor $k_s = 1.05$, while cubic particles of crushed sand and crushed limestone (Figs. 1c, d) are characterised by the value of $k_s =$

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Material type	Average particle diameter d _c , μm	Particle shape factor, k _s	Particle distribution constant, G _{pc}
Cement	22.6	1.52	8.1
Aleuropelite	17.5	2.33	8.1
Fly ash	26.5	1.05	7.6
Crushed sand	27.2	1.65	9.9
Crushed limestone	40.3	1.46	8.9

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Table 4 Numerical criteria for the evaluation of fillers

1.46–1.65, which is close to the value of the shape factor for Portland cement particles $k_s = 1.52$.

The average particle diameter of crushed sand and limestone is correlated with its value for Portland cement $(d_c = 22.6 \text{ m})$. The average size particle of aleuropelite is 1.3 times smaller and average size particle of crushed limestone 2 times larger than size of Portland cement particle.

Rheological behavior of mixtures under squeezing. The plastic behavior of mixtures is characterised by the experimental curves " $F^* - h_i/R$ " (Fig. 2). The experimental curves obtained can be divided into two types. The first

type has a plastic deformation section between two inflection points in the range of relative strains of the sample $0.6 \le h_i/R \le 0.9$, but no pronounced transitions between the curve sections are recorded.

Under the action of low squeezing stress the structure of viscoplastic mixtures remains stable ("placing phase" [26]). That is the first section of the experimental curves "F* – h_i/R " (0.9 ≤ $h_i/R \le 1.0$). When the stresses increase at the second section 0.6 ≤ $h_i/R \le 0.9$, the system deforms plastically ("perfect plastic response phase" [26]). An increase in stress and intensification of flow in the third section are associated with complete destruction





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System ID	К _i (I), кПа	σ ₀ , kPa	σ _{pl} , kPa	$\Delta_{pl}, mm/mm$
C (reference)	2.14	0.45	21.1	0.07
C+Al	3.30	0.38	20.8	0.07
C+FA	3.83	0.44	23.1	0.07
C+CS	3.67	0.33	20.5	0.07
C+CL	5.27	0.32	19.6	0.05

Table 5Rheological properties of 3D-printable mixtures

of the visco-plastic structure ($0.6 \ge h_i/R$). The curves "F* - h_i/R " of this type are typical of all investigated mixtures, with the exception of the mixture containing aleuropelite. The presence of a horizontal section on the curves indicates the ability of viscoplastic systems to plastic deformation without destruction of the structure in a wide range of compressive stresses F* = 2.5 - 15 kPa. The value of plasticity limit estimation for these systems is K_i(I) = 2.14 - 5.27 kPa (Table 5).

The reference system without fillers is characterised by the lowest value of the $K_i(I) = 2.14$ kPa. When fillers are added, the yield stress value increases and the plasticity of the mixtures decreases. The system based on crashed limestone (C +CL) is characterised by the yield stress value $K_i(I) = 5.27$ kPa, i.e. it is the most rigid. The second inflection point in the curve " $F^* - h_i/R$ " at $h_i/R = 0.6$ for the system with aleuropelite filler (C+Al) is not fixed. This means that the structure of the viscoplastic mixture is irreversibly destroyed at the first moment of loading. As a result, it loses stability and becomes fluid. Such systems do not have the necessary viscoplastic properties and stability for extrusion.

The shape stability of the mixtures was evaluated by the results of the analysis of the curves "load P – displacement Δ " (Fig. 3), which shows that all the investigated systems have similar values of structural strength $\sigma_0 = = 0.32 - 0.45$ kPa and plastic strength $\sigma_{pl} = 19.6 - 23.1$ kPa (see Table 5). All systems are characterised by minimal plastic deformation ($\Delta_{pl} < 0.07$ mm/mm) under load.



Fig. 3. Tested 3D-printable mixtures "load P – displacement Δ " curves

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The system C+CL shows the lowest values of structural σ_0 and plastic σ_{pl} strength, and the lowest plastic strains Δ_{pl} (see Table 3, Fig. 3). As a result, the interval between the first crack appearance and fracture is reduced, which indicates the lower stability of the system to the load.

DISCISSION

By evaluating the effectiveness of the influence of numerical filler criteria on the rheological behavior and properties of 3D printable mixtures, it was possible to establish that.

In experimental conditions modelling the visco-plastic flow of mixtures during extrusion (under dynamic conditions), the main factor in ensuring their necessary plasticity and aggregation stability is the kinetic factor, associated with the ability of the dispersed phase to float in the dispersion medium and counteract the kinetic energy of external forces and gravity. This requires maintaining a certain critical size of the dispersed phase particles and their high packing density.

This theoretical position is supported by the experimental results. It was found that the most significant factor in regulating the rheological behavior and plasticity parameters of the mixturess is the filler particles size (Fig. 4). The yield stress value $K_i(I)$ increases by 1.7 times and the plastic deformations decrease by 1.4 times when the average size of the filler increases in the range $d_c = 17.5-40.3 \mu m$. That is to say, there is a directly proportional dependence of the decrease in plasticity of the system with increasing filler



Fig. 4. Influence of filler particle size (a) and shape (b) on 3D-printable mixtures plasticity



particle size. It is important to emphasise that the ratio of binder (cement) to filler particle size is the determining factor. The systems C+Al, C+FA, C+CS, in which the average filler particle diameter is smaller than or approximately equal to the average cement particle diameter ($d_c = 22.6 \,\mu$ m), are characterised by an increase in the yield stress value compared to the reference system without fillers was 1.5–1.7 times, and of 2.6 times for the system C+CL ($d_c = 40.3 \,\mu$ m) it was 2.6 times.

No clear patterns of filler shape influence on the rheological behavior and plasticity of the mixtures were found (see Fig. 4a).

From this it can be concluded that the rheological behavior of mixtures, their plasticity and structural stability under dynamic extrusion conditions is mainly determined by the size and shape of the dispersed phase particles. A necessary condition for the plasticity and aggregation stability of mixtures is the creation of a dense spatial packing of the dispersed phase particles. This is achieved when the filler particles are of a size and multi-size gradation comparable to that of the cement particles.

It has been shown that the rheological behavior and the shape stability of the mixtures are practically independent of the size and shape of the filler particles by simulating the behavior of the mixture under static conditions of layer-by-layer casting. The behavior of heterogeneous microdisperse systems (particle size d ~ 1 100 μ m [21]) under static conditions, such as those studied, is determined by the action of gravitational forces (sedimentation factor) and forces of internal interactions (surface phenomena, contact interactions). Particles assemble into spatial structures when the field of action of these forces is equivalent to that of gravity.

The main factor in the stability of the visco-plastic structure under these conditions is hydrodynamic, which determines its dependence on the density and viscosity of the dispersion medium. Their increase reduces the mobility of the disperse phase particles in the dispersion medium and increases the stability of the system. These parameters for the investigated systems were optimised on the basis of previous studies by introducing a complex viscosity modifier "xanthan gum + liquid glass" in an optimal dosage. In this case, the liquid glass, due to its chemical nature, favours the modification of the ionic composition and the viscosity of the dispersion medium, while the xanthan gum particles increase its viscosity and density. As a result, the main factor determining the rheological behavior of mixtures and their shape stability under static conditions of layer-by-layer casting are the properties of the dispersion medium, regulated by the type and concentration of viscosity modifiers.

Therefore, within the design of 3D printable mixtures, the main criteria of fillers are the particle distribution constant G_{pc} , which characterises the filler particle size gradation, and the average particle diameter d_{pc} .

CONCLUSION

The workability of 3D-printable mixtures is determined by their plasticity and structural stability under load during extrusion and layer-by-layer casting.

It has been found that, the creation of a dense spatial packing of disperse phase particles is a necessary condition for the plasticity and aggregation stability of mixtures under dynamic extrusion conditions. Within the design of 3D printable mixtures, the most important properties of fillers have been established:

- the average diameter of filler particles dc, which should not exceed the average diameter of cement particles;
- the particle distribution constant G_{pc} , which characterises the particle size distribution. The rational range of it value $G_{pc} = 7.5-10$ corresponds to the multi-size gradation of the filler particle.

The properties of the fillers are not decisive for the shape stability of the mixtures under the static conditions of layer-by-layer casting. The primary factor is the properties of the liquid phase (dispersion medium), which is regulated by the type and dosage of viscosity modifiers.

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Galina S. Slavcheva – scientific leadership; research concept; development of methodology and research methods; scientific editing of the article.

Valentina A. Solonina - conducting experimental studies; analysis of experimental data; drawings for the article.

Igor O. Razov – mathematical description, performance of analytical calculations.

Pavel V. Filipenko - experimental studies.

Viktor S. Orlov – experimental studies.

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