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Application of monosaccharides derivatives in colloidal processing of aluminum oxide

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Abstract

Obtaining highly loaded, time-stable and relatively low viscosity suspensions approaches colloidal processing to be very convenient and effective route of shaping of nanopowders. In order to obtain well dispersed, homogenous ceramic slurries, certain additives are given. Saccharides, particularly monosaccharides, as well as their derivatives, were found to be a group of effectively working processing agents in case of alumina, which has been used as a solid phase of highly loaded nanosuspensions. This class of chemical compounds can be described by a series of advantages – they are non-toxic, water-soluble, inexpensive, etc. In this paper suspensions of nano- and submicro-alumina powders with addition of D-fructose, 1-O-methyl-D-fructose, D-glucose and 3-O-acrylic-D-glucose have been studied in terms of their rheological properties, moreover the properties of as-received green bodies have been presented.

Keywords: Monosaccharides; Al_2O_3; Nanopowders; Rheological properties; Colloidal processing

1. Introduction

Shaping of nanopowders puts high demands on present-day science and technology. Their high surface energy hinders such traditional forming methods like, e.g. die pressing. To meet challenges of nanopowders shaping, researchers are intensively looking for some new forming methods, which could be useful for this specific sort of powders. Very promising for this application seems to be colloidal processes.

In recent decades, many novel shaping technologies have been developed on the basis of colloidal processing of ceramic powders, for example slip casting,[1] gelcasting,[2,3] monomer based sol–gel,[4,5] electrophoretic deposition[6,7] or direct coagulation casting.[8] Colloidal processing of high performance ceramics requires new, effectively working processing agents like deflocculants,[9] binders,[10] organic monomers,[11,12] etc. The mentioned components can ensure advantageous microstructure and good mechanical properties of as-obtained ceramics; moreover they facilitate the processing route. The main challenge of colloidal processing lies in achieving time-stable, low viscosity dispersions having high solid loading of well dispersed particles. Processing agents have to influence these desirable properties of ceramic suspensions as well as they should realize the trend of "green chemistry".

In recent years monosaccharides and their derivatives have been found to lie in area of researchers' interest. Monosaccharides, as processing agents, have many advantages. Firstly they are non-toxic, water-soluble, inexpensive and readily available. Monosaccharides can be easily removed from samples during binder burnout process and they have the influence on high mechanical strength of green bodies. Application of monosaccharides (mainly D-fructose) as dispersing agent was first described by Schilling et al.[13] and Li et al.[14] They found that molecules of D-fructose adsorbs on a surface of alumina nanoparticles and displace adsorbed water molecules. This displacing increases the amount of free (bulk) water in suspension and makes an effective nanoparticle radius smaller. Therefore, layers of water that surround ceramic particles cannot interact, what results in decreasing the viscosity of nanosuspensions.

Other conducted studies showed that derivatives of monosaccharides can be used as additives, namely organic monomers in gelcasting process.[15] Gelcasting is the shaping method that combines conventional moulding from slips with polymer chemistry. It allows obtaining high-quality, complex-shaped ceramic elements by means of an *in situ* polymerization, through which

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a macromolecular network is created to hold ceramic particles together.

The paper discusses applications of monosaccharides and their derivatives as dispersing agents for alumina suspensions. The greatest emphasis has been placed on the influence of chemical structure of applied compounds on rheological properties of nanoalumina suspensions with addition of D-fructose and its derivative 1-O-methyl-D-fructose. The properties of green bodies obtained by slip casting are also presented. Furthermore, the dispersing properties of 3-O-acrylic-D-glucose (initially synthesized as monomer for gelcasting) for nano- and submicro-alumina are shown.

2. Experimental procedures

2.1. Materials

The research has been carried out for two alumina powders. The first one was Al_2O_3 NanoTek® (Alfa Aesar, Germany), the same type of nanosized alumina that was used by Schilling at al.[13] and Li et al.[14] The powder was a mixture 70:30 of δ-Al_2O_3 and γ-Al_2O_3 phases with a density 3.53 g/cm^3 measured on AccuPyc II 1340 Pycnometer (Micromeritics, USA), a specific surface area 35.0 m^2/g measured by BET method and the average particle size of 47 nm calculated from BET. The particles were spherical in shape, agglomerated and were used as received from the supplier. The second powder was high purity α-Al_2O_3 TM-DAR (Tamei Chemicals, Japan) of an average particle size 0.21 μm (calculated from BET), density 3.80 g/cm^3 measured on AccuPyc II 1340 Pycnometer (Micromeritics, USA) and a specific surface area 14.1 m^2/g. As dispersing agent the following monosaccharides and their derivatives have been used: D-fructose (POCh, Poland), 1-O-methyl-D-fructose, D-glucose (POCh, Poland), 3-O-acrylic-D-glucose (Fig. 1). Monosaccharides derivatives have been synthesized by authors. Basing on rheological measurement it has been shown in the previous research[16,17] that the positions and orientations of hydroxyl groups in a molecule influence the ability of monosaccharide to disperse nanometric-alumina suspensions. Therefore, the change of a chemical structure of monosaccharides should improve their influence on the viscosity of suspensions. To change the deflocculating properties of D-fructose one of the hydroxyl groups was substituted by $-OCH_3$. In this way it was possible to obtain 1-O-methyl-D-fructose. This fructose derivative was made in a three step synthesis according to the procedure described by Glen et al.[18] 3-O-acrylic-D-glucose was synthesized in a three step synthesis elaborated by authors, described elsewhere.[19]

2.2. Preparation of ceramic suspensions and shaping

Research methodology was conducted by two paths. Path I was carried out for nanometric-alumina with application of D-fructose and 1-O-methyl-D-fructose as effective dispersing agents. Path II was carried out for nanometric and submicro-alumina with application 3-O-acrylic-D-glucose as organic monomer able to polymerize *in situ* in shaping ceramic powders by gelcasting methods. During the research it became clear that this compound plays also the role of dispersing agent for nanometric and submicro-alumina, which is presented in this paper. That is why the dispersing properties of 3-O-acrylic-D-glucose were compared to D-glucose. The division into two experimental paths was dictated by peculiar characteristics of synthesized monosaccharides derivatives. 3-O-acrylic-D-glucose contains double bond between carbons in a molecule; therefore all activities with ultrasonification had to be eliminated. It was due to uncontrolled and undesired polymerization caused by the increase of temperature related with application of ultrasonification. Even normally applied cooling of suspensions could not prevent the polymerization because the locally overheating in the neighborhood of immersed tip of the ultrasonicator was high enough to initiate polymerization.

2.2.1. Path I

Nanometric-alumina aqueous suspensions with monosaccharide additive were prepared in redistilled water at room temperature. In each experiment the solid content in the suspensions was 30 vol.%. The concentration of monosaccharide was varied from 1 to 5 wt.% (based on the alumina powder). Suspensions were mixed in alumina container in a planetary ball mill PM100 (Retsch) for 90 min with a speed of 300 RPM. Subsequently, the alumina aqueous suspensions were ultrasonificated (Model 3000 Ultrasonic Homogenizer, Biologics, Inc.) for 15 min and later mixed for 15 min once again in the planetary ball mill. Rheological properties were measured using Brookfield DV + II-Pro rheometer (Brookfield Engineering Laboratories Inc., Massachusetts, USA). The shear rate increased from 0.1 to 100 s^{-1} and back to 0.1 s^{-1}. The rheological measurements were carried out with spindle S18. Then the specimens were formed by slip casting method on porous alumina substrate in polyethylene moulds. The density of obtained green bodies was measured by Archimedes' method in kerosene. The microstructure of green specimens was observed in scanning electron microscope SEM JEOL JSM-6500F.

2.2.2. Path II

Alumina aqueous suspensions with saccharide additive were prepared in redistilled water at room temperature. The solid contents for NanoTek alumina were 30 and 35 vol.%, while for TM-DAR alumina it was 40 vol.%. The concentration of monosaccharide was 3 wt.% (based on the alumina powder) for NanoTek alumina and 5 wt.% for TM-DAR alumina. The previous research showed that saccharides cannot be used as dispersing agents for micrometric alumina powders while for nanopowders they work efficiently.[13] It is seems that it is not surface area but size of particles that determines the application of such compounds as dispersing agents. Probably it is due to the differences in distances between the particles in the nano- and micrometric alumina suspensions.[14] Probably, that is why, to deflocculate the submicrometric alumina TM-DAR (210 nm) it was necessary to use higher amounts of saccharides then for nanopowder. Suspensions were mixed in alumina container in a planetary ball mill PM100 (Retsch) for 90 min with a speed of 300 RPM. Then rheological properties were measured using

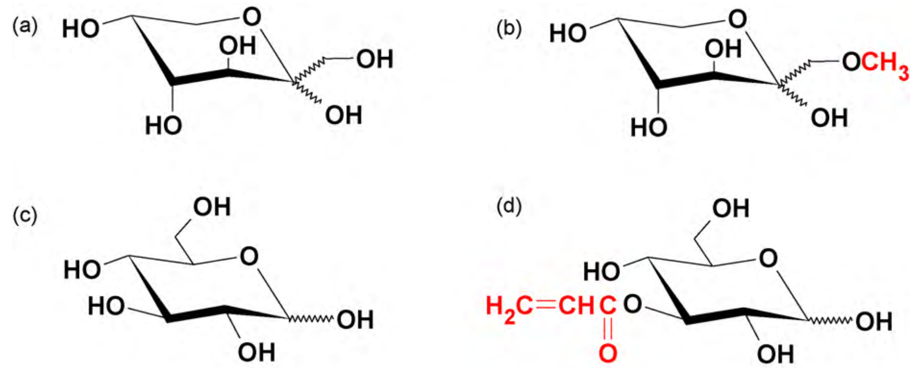

Fig. 1. Molecular structure of (a) D-fructose, (b) 1-O-methyl-D-fructose, (c) D-glucose and (d) 3-O-acrylic-D-glucose.

Brookfield DV + II-Pro rheometer (Brookfield Engineering Laboratories Inc., Massachusetts, USA). The shear rate increased from 0.1 to 35 s^{-1} and back to 0.1 s^{-1}. For this measurement a spindle S34 was used.

Zeta potential measurements in this study for all suspensions were conducted on a Zetasizer 3000 (Malvern Instruments). The saccharides were added to nanoalumina solution where concentration of alumina was about 100 ppm. The concentration of NaCl electrolyte in the solution was 10^{-3} mol/dm^3. The solution was ultrasonicated for 5 min before the measurements. The pH of the suspension was adjusted using 0.1 mol/dm^3 HCl or NaOH solution and varied from 2 to 11.

3. Results and discussion

3.1. Zeta potential

Fig. 2a shows the change of the zeta potential of nanoalumina (NanoTek) suspension as a function of pH in the presence of D-fructose and 1-O-methyl-D-fructose and without any addition of the saccharide. One can see that the isoelectric point (IEP) of pure nanosized alumina in the present investigation can be found at pH 9.2. The measurements showed that the addition of saccharide does not shift the isoelectric point and only slightly changes the value of zeta potential in comparison to suspension without any addition. It also can be noticed that the higher concentration of saccharides the higher value of zeta potential. An example of such situation was showed in Fig. 2b, where the increase of D-fructose concentration from 1 to 5 wt.% slightly increases the value of zeta potential. High value of the zeta potential of examined alumina suspensions (70 mV and above for pH <7) indicate that such suspensions have high stability. Fig. 2c shows the change of the zeta potential of submicro-alumina (TM-DAR) suspension as a function of pH in the presence of 3-O-acrylic-D-glucose and without any addition of saccharide. Similarly as for the other mentioned saccharides the addition of these compounds does not shift the isoelectric point.

Saccharides are neither electrolytes nor polymers. For example, the pK_a value of D-fructose and 1-O-methyl-D-fructose is 12.03 and 11.52 respectively. It means that in a wide range of pH such saccharides do not dissociate. Because the pH of pre-

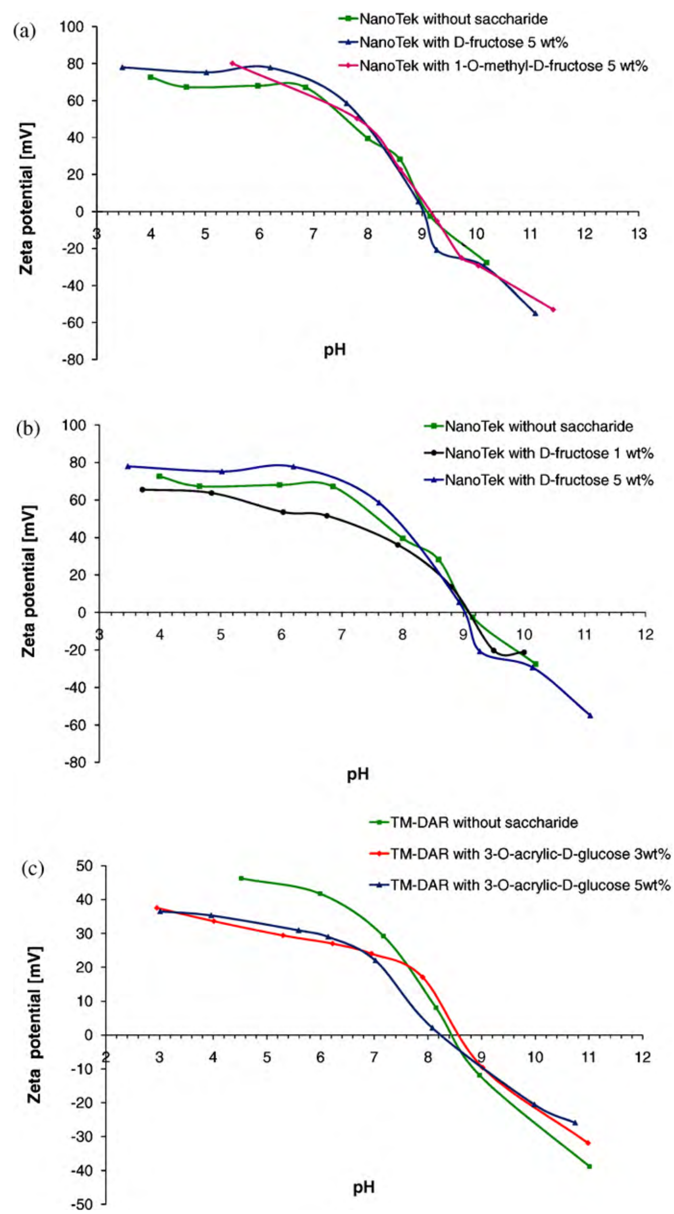

Fig. 2. Zeta potential curves of alumina suspensions: (a) NanoTek without any addition and with 5 wt.% of D-fructose and 1-O-methyl-D-fructose; (b) NanoTek with 1 and 5 wt.% of D-fructose; (c) TM-DAR without any addition and with 3 and 5 wt.% of 3-O-acrylic-D-glucopyranose.

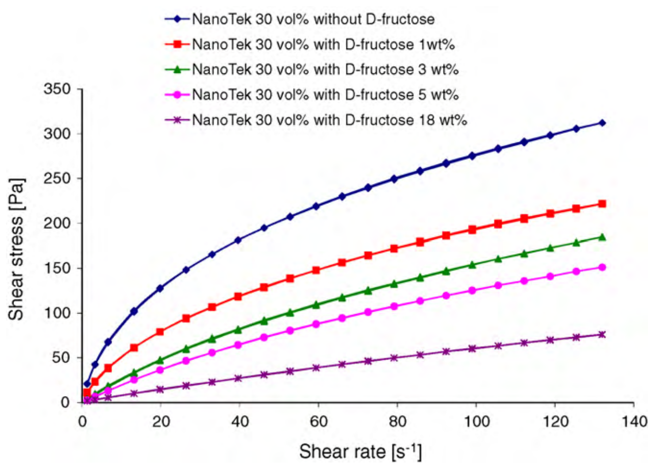

Fig. 3. Shear stress in function of shear rate for NanoTek alumina suspensions with different concentration of D-fructose.

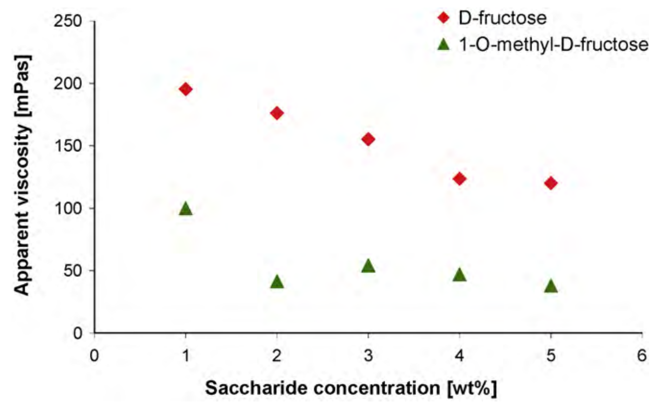

Fig. 4. Apparent viscosity in function of shear rate of NanoTek alumina suspensions with different concentration of D-fructose and 1-O-methyl-D-fructose.

pared suspensions was about 5–7, hence in such conditions the saccharides exist as undissociated. That is why they do not have an effect on shifting the IEP.

Basing on zeta potential measurement it can be stated that saccharides do not change the charge density on the alumina particles surface which means that the saccharides do not affect the double-layer repulsion.

3.2. Suspensions characterization

Fig. 3 shows the shear stress curves of NanoTek alumina suspensions with different concentration of D-fructose at different shear rates, where shear stress increases with the increase of shear rate. Additionally the suspensions exhibit shear thinning behavior without the initial resistance to deformation. This type of behavior is known as pseudoplastic and indicates that such suspensions are weakly flocculated. In Fig. 3 it can be seen how the increasing concentration of D-fructose in the suspension changes the appearance of the curves. These curves can be described by Herschel–Bulkley's equation $\tau = \tau_0 + K(\gamma)^n$ which enables to calculate the shear stress (τ), yield stress (τ_0), the flow index (n) and consistency index (K) at shear rate (γ).

Table 1
Flow indexes and consistency indexes calculated for NanoTek alumina suspensions with solid content of 30 vol.% with varying concentration of D-fructose and 1-O-methyl-D-fructose. Calculations were made according to Herschel–Bulkley's model.

Saccharide concentration (wt.%)	D-Fructose		1-O-methyl-D-fructose	
	n	K (mPa s)	n	K (mPa s)
0	0.56	2204	0.56	2204
1	0.63	1112	0.77	294
3	0.83	352	0.88	97
5	0.87	244	0.91	57
18	0.88	108	–	–

Where n and K are flow index and consistency index respectively according to Herschel–Bulkley's model.

The calculated values of n and K for NanoTek alumina with D-fructose and 1-O-methyl-D-fructose are shown in Table 1. One can see that the higher D-fructose concentration in the suspension the lower value of K parameter, which means that viscosity is decreasing. Decreasing slope of the graph with increasing saccharide concentrations informs that the suspensions become less and less flocculated. The increasing value of the flow index with increasing D-fructose concentration indicate that the suspensions behavior is changing from pseudoplastic to Newtonian-like behavior (n tends to 1). The similar character of rheological behavior was found for 1-O-methyl-D-fructose. These results are in good agreement with previous results obtained by Akinc and Li.[14] Their research shows that viscosity of alumina suspension (30 and 40 vol.% of solid content) decreases steadily with the increase of D-fructose concentration up to 15 wt.%.

The substitution of one hydroxyl group by $-OCH_3$ in D-fructose molecule significantly improved flow behavior, what is shown in Fig. 4. For the same concentration of saccharides the viscosity of suspensions with 1-O-methyl-D-fructose is ca. 100 mPa s lower in comparison to the viscosity of suspensions with D-fructose. A closer look at Table 1 reveals that for some concentration of saccharides in the suspensions the calculated flow index and consistency index for suspension with 1-O-methyl-D-fructose are higher (tends to 1) and smaller respectively than for the suspensions with D-fructose. The particle volume fraction in all cases was 30 vol.%. The differences in dispersing properties between D-fructose and 1-O-methyl-D-fructose can be explained by the ability of saccharides to decrease the water layers around the alumina particles.[13] Greater ability of 1-O-methyl-D-fructose to deflocculate the nanosized alumina suspension can be related to compatibility of saccharide in three-dimensional hydrogen-bonded structure of water. Galema et al.[20] stated that the methylhexopyranosides, like 1-O-methyl-D-fructose, are less compatible (fit worse) with the three-dimensional hydrogen-bonded structure of water than hexoses like D-fructose. Due to the presence of an additional methoxy group, methylhexopyranosides disturb more water molecules than the hexoses. In this situation it is possible that the same concentration of 1-O-methyl-D-fructose releases more water molecules from surface and decreases the thickness of

Table 2
Flow indexes and consistency indexes calculated for TM-DAR and NanoTek alumina suspensions with 3 and 5 wt.% of D-glucose and 3-O-acrylic-D-glucose. Calculations were made according to Herschel–Bulkley's model.

Alumina powder	Solid content (vol.%)	Saccharide concentration (wt.%)	D-Glucose		3-O-acrylic-D-glucose	
			n	K (mPa s)	n	K (mPa s)
NanoTek	30	0	0.22	17,421	0.22	17,421
NanoTek	30	3	0.23	15,911	0.44	4022
NanoTek	35	3	Paste-like		0.49	3329
TM-DAR	40	0	0.37	3884	0.37	3884
TM-DAR	40	5	0.34	2751	0.70	243
TM-DAR	50	5	Paste-like		0.41	8936

Where n and K are flow index and consistency index respectively according to Herschel–Bulkley's model.

water layers more than D-fructose. Higher disorder of water layers, caused by 1-O-methyl-D-fructose, may increase the amount of free (bulk) water in suspensions, increase water molecules mobility and decrease the viscosity of the suspensions in the higher extent.

The calculated values of n and K for NanoTek and TM-DAR alumina with D-glucose and 3-O-acrylic-D-glucose are shown in Table 2. One can see that it was impossible to obtain slurries of viscosity low enough to keep flow properties for NanoTek 35 vol.% and TM-DAR 50 vol.% with D-glucose, while the application of 3-O-acrylic-D-glucose allowed obtain slurries of high solid loading and satisfactory viscosity that allows effectively cast these slurries into moulds. Similar to D-fructose, the presence of 3-O-acrylic-D-glucose causes the decrease of suspension viscosity (the lower value of K parameter). The value of the flow index is increasing when the saccharide derivative is added to the slurry, but the growth is not as significant as for D-fructose. It may be caused by a different way of preparing ceramic slurries, described in Section 2.2 as Path II, where the main difference it that in case of 3-acrylic-D-glucose no activities with ultrasonification were carried out.

Fig. 5a shows the flow curves for nanometric NanoTek alumina with two saccharides: D-glucose and 3-O-acrylic-D-glucose. Once can see that the application of D-glucose decreases only slightly the viscosity of 30 vol.% slurry comparing to the slurry without any addition of saccharide. Whereas the substitution of one hydroxyl group by acrylic group in D-glucose molecule significantly improved flow behavior of NanoTek alumina of 30 vol.% solid content in the slurry. The application of 3 wt.% of D-glucose to the slurry of 35 vol.% solid content results in obtaining paste-like slip, while the same concentration of 3-O-acrylic-D-glucose allows slurry to flow, similar to those of 30 vol.% solid content with the same saccharide derivative.

The similar tendency was observed for submicro-TM-DAR alumina what is shown in Fig. 5b. Prepared slurry of 40 vol.% without any addition of saccharide exhibit flow limit, while the addition of 5 wt.% of 3-O-acrylic-D-glucose results in obtaining slurry of over ten times lower viscosity. The application of the same concentration of D-glucose results in obtaining dispersed suspension but of visibly higher viscosity than for 3-O-acrylic-D-glucose. The increase of TM-DAR solid content to 50 vol.% results in obtaining paste-like slurry with application of 5 wt.% of D-glucose and well dispersed slurry with application of 5 wt.% of 3-O-acrylic-D-glucose, what is shown in Table 2.

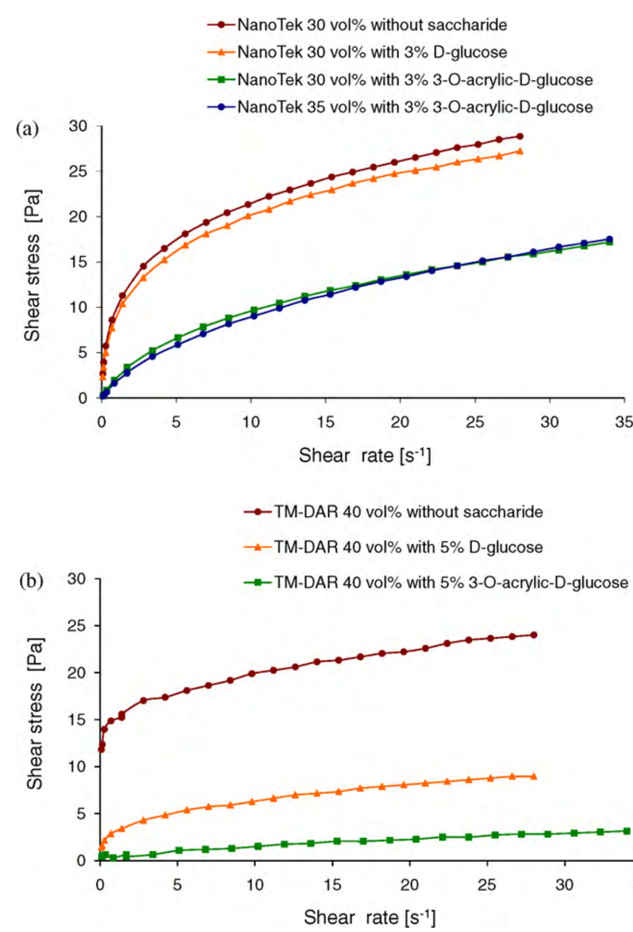

Fig. 5. Shear stress in function of shear rate for (a) NanoTek alumina suspensions with 3-O-acrylic-D-glucose; (b) TM-DAR alumina suspensions with 3-O-acrylic-D-glucose.

3.3. Characterization of green bodies

Application of saccharides as processing additives allows obtaining the suspensions with low viscosity and high stability. For that reason such suspensions can be used in slip casting. Moreover it is possible to obtain green bodies of high density.

Table 3
Densities of green bodies obtained by slip casting with application of different concentration of D-fructose and 1-O-methyl-D-fructose.

Saccharide	Concentration (wt.%)	Relative density (%)
Without	0	60
D-Fructose	1	63
D-Fructose	3	66
D-Fructose	5	66
1-O-methyl-D-fructose	1	66
1-O-methyl-D-fructose	3	68
1-O-methyl-D-fructose	5	69

The effect of D-fructose and 1-O-methyl-D-fructose on green density is presented in Table 3. Generally speaking, the addition of saccharide to the suspensions increases the density of green bodies. For the same concentration of 1-O-methyl-D-fructose and D-fructose in the nanosized alumina suspensions, samples obtained with 1-O-methyl-D-fructose used as dispersing agents have higher green densities. It must be noticed that in all cases obtained green densities are unusually high. Such high density of green bodies is rather typical for samples obtained by pressing. The explanation of this can be found if we look closer to the particle size distribution and to the morphology of nanometric-alumina. The particles are spherical in shape. Although the average particle size is 47 nm, the real particle size is ranging from 10 to 100 nm. Due to the low viscosity of suspensions the nanometric-alumina particles can move and arrange easily during the moulding of green bodies. Smaller particles can fill the empty spaces between bigger ones and give high packing density, which in consequence results in high green density. Therefore, the obtained density is higher than for samples made from spherical particles of equal size. The arrangement of particles is confirmed by SEM image presented in Fig. 6.

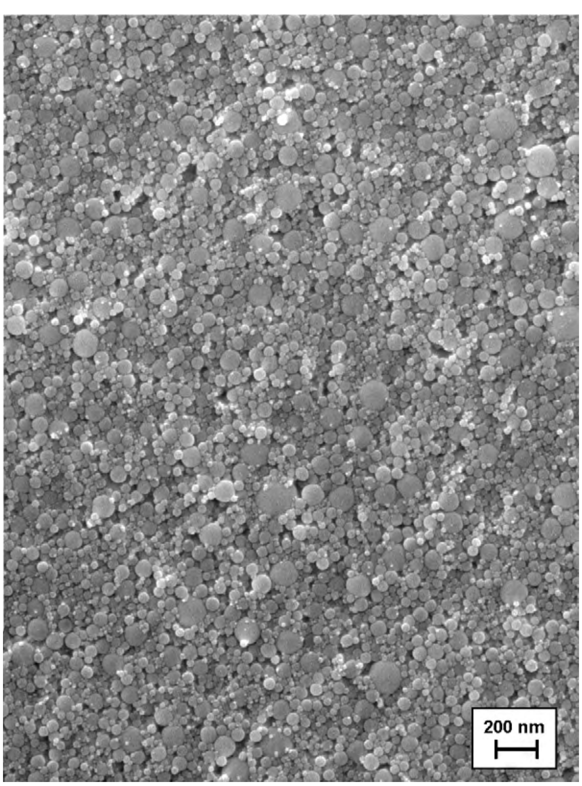

Fig. 6. SEM microstructure of NanoTek green body obtained with application of 1-O-methyl-D-fructose.

4. Conclusions

Application of saccharides makes it possible to obtain nanometric and submicro-alumina suspensions with low viscosity and high stability. The selected monosaccharides as well as both presented derivatives do not result in shifting isoelectric point of alumina. It also means that the potential mechanism of saccharide in reducing the viscosity is not based on electrostatic stability.

The substitution of $-OCH_3$ for OH group increases the deflocculating properties of D-fructose. Probably, it is due to lower compatibility of 1-O-methyl-D-fructose with the three-dimensional hydrogen-bonded structure of water. Therefore 1-O-methyl-D-fructose molecules can weaken the interaction between the particle surfaces and water molecules more then D-fructose, as a consequence, they release more bound water layer. It makes the suspensions with 1-O-methyl-D-fructose less viscous than the suspensions with D-fructose. Moreover, the synthesized low-toxic compound 3-O-acrylic-D-glucose, initially obtained as organic monomer for gelcasting process, can play double role: as monomer able to polymerize in situ as well as a dispersing agent for nano- and submicro-alumina suspensions. Thanks to low viscosity and high stability of suspensions with saccharides, it is possible to obtain the green bodies of high density.

Due to organic synthesis it is possible to tailor the structure and consequently the properties of processing additives to the shaping methods. This makes the organic synthesis becomes a powerful tool for obtaining new environmental friendly processing additives for colloidal processing.

Acknowledgements

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