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Rheological behaviour and mechanical properties of porcelain stoneware bodies containing Italian clay added with bentonites

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Abstract

This paper presents the results obtained by replacing German and Ukrainian clays in a porcelain stoneware body with a clay from Sardinia island, Italy, added with 10 wt% of different bentonites available on Italian territory in order to lower the body costs. Two series of bodies were prepared: a first series by substituting completely German clay and partially Ukrainian clay (5 wt%) and a second series by substituting completely the Ukraine clay. Rheological measures were carried out on the suspensions prepared in order to verify the effect of the bentonites presence on the shear and time dependence. The fired specimens were characterized across the technological parameters (water absorption, linear shrinkage, etc.), mechanical and aesthetical properties and compared with a standard/industrial composition.

Keywords: C. Mechanical properties; D. Traditional ceramics; Bentonites; Rheological properties

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1. Introduction

The porcelain stoneware tile is a product with high aesthetical qualities and very good technical characteristics obtained by fast single firing (35–45 min) at about 1200–1230 °C. It is used in large quantities and in increasing application fields. The Italian production of this typology was 383 million of square meters in 2006 corresponding to 67% of the total tile national production [1]. The porcelain stoneware tile shows specific characteristics: extremely low open porosity, water absorption ($WA\% \leq 0.05\%$), high abrasion resistance and improved mechanical properties [2–4].

Porcelain stoneware bodies are primarily composed of plastic and kaolinitic clays, sodium and potassium feldspars and quartz, heat treated to form a mixture of glass and crystalline phases. The clay minerals are the main components (40–55 wt%) of the ceramic body. They have an important function: to confer plasticity and workability in the green state and, during firing, undergo some structural modifications which furnish the main oxides to form some important phases such as mullite ($3Al_2O_3{\cdot}2SiO_2$). Generally, Italian clays are not suitable for porcelain stoneware tiles production because of their high content of chromophore oxides and low plasticity. In fact, nowadays it is necessary to import clays from foreign countries such as Ukraine, Germany, France and Portugal (about 3×10^6 ton year^{-1}) [5,6] which provide high breaking strength in green, low expansion post pressing and lower amounts of iron and titanium oxides. The aim of this paper was to prepare new clayey mixtures with similar features and costs lower than the foreign raw materials, by using a Sardinian clay added with 10 wt% of different Italian bentonites. Bentonites were used in this study to improve the plasticity of the Sardinian clay in the green state.

Bentonites are expanding type sheet silicates constituted of one octahedral layer between two tetrahedral layers with a high cationic exchange capacity (Na, K, Ca, and Mg) and specific surface; they can also absorb interlayer water molecules increasing the basal length. Their composition consists mostly of montmorillonite clay (at least 70 wt%) [7]. The mixture of bentonite and water forms colloidal suspensions with plastic behaviour (presence of yield stress) and thixotropy. For this

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reason it is very important to verify the rheological behaviour of the new clayey mixtures from shear and time dependence point of view in order to find the typologies that do not provoke dangerous effects on the ceramic process.

2. Experimental procedure

For this study a Sardinian kaolinitic clay added with 10 wt% of different bentonites available on the Italian territory was used to prepare a new clayey mixtures. Fourteen bentonites were characterized across the measure of the elastic modulus (E) and breaking strength (BS) in order to choose six samples with the best mechanical properties.

The elastic modulus values were determined using a non-destructive impulse excitation technique (Lemmens Grindosonic Electronika MK5, LTD). This test meets the ASTM 1259 testing method for dynamic elastic modulus [8]. The breaking strength was determined using a flexometer (Nassetti, Fiorano Modenese, Italy) by a three-point loading test and the values reported by indicating the maximum strength that the sample can bear before breaking were obtained according to UNI EN ISO 10545.4 as the average of six pieces.

The chemical analysis of the bentonites was performed by inductively coupled plasma (ICP-Varian Liberty 200) for the main elements.

The mineralogical analysis of the bentonites samples was carried out by using a conventional Bragg–Brentano powder diffractometer (PW 3710, Philips Research Laboratories) with Ni-filtered Cu Kα radiation. The patterns have been recorded on the ground samples (<25 μm in size) in the 5–70° 2θ range (step size 0.02° and 1 s counting time for each step).

Two series of porcelain stoneware bodies were prepared to compare with a standard one: the first (Series I) was obtained by substituting completely German (G) clay and partially Ukrainian (U) clay (5 wt%) and the second one (Series II) by substituting completely the Ukrainian clay as reported in Table 1. The obtained mixtures have been wet ground (33 wt% of water and 0.3% dry solid of sodium tripolyphosphate deflocculant) for 40 min in a laboratory ball mill by using alumina sintered grinding media, dried overnight at 110 °C, ground and sieved below 63 μm. The dried powders have been humidified to a water content of 7 wt% and uniaxially pressed at 40 MPa to produce round (40 mm diameter, 5 mm thickness) samples (10 g). Moreover, rectangular tiles (60 g) of 100 mm × 50 mm × 8 mm mm were prepared for breaking strength measures. The specimens have been dried overnight at 110 °C and then fired in an electrical roller laboratory kiln at a maximum temperature of 1200 °C with a thermal cycle of 43 min cold-to-cold [9].

The mixture's particle-size distribution was determined by using a laser size analyzer (Master Sizer, Malvern, UK). On the mix suspensions prepared a rheological shear and time dependence analysis was performed. The measurements were conducted at 25 °C, in continuous shear flow by using a Control Rate Searle, viscometer (Haake, VT550), equipped with coaxial cylinder sensor MV1. All the samples were measured with a standard procedure to study the shear-dependent behaviour using four-step cycle: 1 min at a shear rate of 200 s^{-1} to exclude the effects of the sample preparation step; at rest for 1 min; a shear rate increase from 0 to 200 s^{-1} in 10 min, followed by a shear rate decrease from 200 to 0 s^{-1} in 10 min. In order to characterize the time-dependent properties, on–off procedure was applied ($\dot{\gamma} = 100\,\mathrm{s}^{-1}$). On pressed mixture samples (green and dried) breaking strength test was conducted with the same procedure of the bentonites samples.

The bodies' densification was described in terms of linear shrinkage (LS%) and water absorption (WA%) according to UNI EN ISO 10545.3. Linear shrinkage values were obtained for all samples using the following equation:

$$\mathrm{LS}\,(\%) = \frac{L_g - L_f}{L_g} \times 100 \tag{1}$$

being L_g and L_f the diameter of the green and fired specimens, respectively. The LS% values obtained for five specimens were averaged for each composition.

Colorimetric measures by Minolta instrument were performed on the fired samples using CIELab* colour space (Illuminant D65, Observer 10°).

3. Results and discussion

Chemical composition of the 14 bentonite samples studied (Table 2) indicates the alkaline nature of the clay minerals, where the exchangeable cations are normal Na, K, Ca and Mg.

Table 1
Batch composition of the mixtures prepared: (a) GBi: mixtures Series I, (b) UBi: mixtures Series II compared to the reference one (REF)

Mixtures	GB1	GB2	GB3	GB4	GB5	GB6	UB1	UB2	UB3	UB4	UB5	UB6	REF
U clay	18	18	18	18	18	18	–	–	–	–	–	–	23
G clay	–	–	–	–	–	–	13	13	13	13	13	13	13
Turk feld.	32	32	32	32	32	32	32	32	32	32	32	32	32
Italian feld.	20	20	20	20	20	20	20	20	20	20	20	20	20
Sand	12	12	12	12	12	12	12	12	12	12	12	12	12
Sardinian clay	16.2	16.2	16.2	16.2	16.2	16.2	20.7	20.7	20.7	20.7	20.7	20.7	0
B1	1.8						2.3						0
B2		1.8						2.3					0
B3			1.8						2.3				0
B4				1.8						2.3			0
B5					1.8						2.3		0
B6						1.8						2.3	0

Table 2
Chemical analysis (wt% oxide) of the bentonite samples investigated

Element	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10	B11	B12	B13	B14
SiO_2	56.50	62.90	57.50	73.80	60.20	59.50	57.60	72.80	69.40	58.70	62.70	58.10	58.60	58.30
Al_2O_3	23.00	21.00	14.20	12.90	14.70	14.14	14.30	12.1	13.00	17.70	11.70	14.70	17.90	18.60
Fe_2O_3	2.00	1.60	4.76	1.42	5.20	5.29	5.29	2.12	1.26	3.12	1.72	5.05	4.43	5.63
TiO_2	0.19	0.19	0.46	0.13	0.67	0.67	0.63	0.24	0.14	0.52	0.22	0.19	0.71	0.63
CaO	2.40	0.71	3.03	2.14	4.06	3.14	4.82	1.04	2.26	2.53	0.79	3.18	3.64	2.05
MgO	2.20	2.45	5.77	1.30	2.43	2.28	2.36	2.56	3.15	3.63	1.67	3.63	2.97	4.34
K_2O	2.90	2.62	1.45	1.08	3.96	3.78	3.97	0.74	1.35	0.58	0.65	0.75	0.82	0.88
Na_2O	2.90	1.74	0.46	0.46	1.63	4.82	4.11	2.62	2.89	2.83	0.72	3.23	1.42	1.23
LOI	7.40	6.74	12.36	6.72	7.17	6.36	6.81	5.74	6.49	8.05	19.74	10.95	9.43	8.13
Total	99.59	99.95	99.99	99.95	100.6	99.98	99.89	99.96	99.94	99.90	99.91	99.80	99.92	99.80
S	[a]	0.33	[a]	[a]	[a]	[a]	[a]	0.13	0.01	0.04	0.14	0.83	1.01	0.04

[a] $S < 0.01$ (element S (wt%)).

Table 3
Mechanical properties of the bentonites investigated

Bentonite	B1	B2	B3	B4	B5	B6	B7	B8	B9	B10	B11	B12	B13	B14
BS (green) (kgf cm^{-2})	24.24	29.76	17.17	9.29	18.38	22.93	22.67	17.21	12.44	12.77	12.73	19.26	22.31	22.49
BS (dried) (kgf cm^{-2})	95.38	131.25	35.02	24.32	70.08	81.98	75.21	64.54	30.59	16.56	34.65	40.09	53.90	28.06
E (GPa) (green)	3.08	3.02	1.95	1.44	2.52	3.32	3.21	1.98	2.12	2.05	1.86	2.09	2.08	1.62
E (GPa) (dried)	6.99	3.95	2.76	2.98	5.19	6.19	6.47	4.78	4.87	2.45	2.72	3.41	3.12	3.17

The loss of ignition (LOI) is around 10% for all samples except in B11 that presents the highest value (~20%) related to the loss of interlayer water molecules (~180 °C) and to dehydroxilation phenomenon (450–650 °C). Besides, it is important to note the iron oxide content of the samples for their use into a porcelain stoneware body: the bentonites having a $Fe_2O_3 > 5\%$ will not be used to avoid the black core and changes on colorimetric properties of the ceramic tile after firing.

The data regarding mechanical tests (BS and E) are reported in Table 3. Six bentonites from the 14, having the best agreement between the mechanical and chemical–mineralogical characteristics in green and in dried samples were chosen. For example, the B5, B6 and B7 samples show good values of BS and E but because of the high-iron oxide content they cannot be used in the ceramic body. The samples B1, B2, B8, B10, B13 and B14 show good mechanical properties and a suitable composition for porcelain stoneware body: B10 sample has not high BS and E values but was chosen for the high-montmorillonite content. The selected bentonite samples were recalled as B1 = B1, B2 = B2, B8 = B3, B10 = B4, B13 = B5, and B14 = B6.

Table 4
XRD mineralogical analysis of the bentonites used

Sample	Crystalline phase
B1	Quartz, syn, albite, ordered, microcline, illite, kaolinite, montmorillonite
B2	Quartz, albite, illite, montmorillonite kaolinite, microcline
B3	Cristobalite low, montmorillonite, quartz, albite high
B4	Montmorillonite, quartz low, calcite kaolinite, hematite
B5	Montmorillonite-15A, quartz, calcite, calcium sulfate, albite high
B6	Montmorillonite, quartz low, hematite, albite disordered, anorthite, illite

The mineralogical composition of the bentonites chosen has been studied by X-ray diffraction analysis (Table 4). The patterns of the samples present typical spectra of a standard bentonite, with a presence of clay minerals from the smectite group. All the samples present a peak at 3.34 Å, characteristic of the presence of the quartz, samples B3 and B5 show the presence of low cristobalite too. Montmorillonite is present in all samples; in particular B4 sample has the highest intensity peak (as shown in Fig. 1). It is also important to note that B1 and B2 samples show the presence of kaolinite (which is not so plastic as the smectite group clays) and are composed by illite too, which normally increase the plastic behaviour of a clay mixture. The good mechanical properties of these samples are probably related to the content of clay minerals. Besides, B4 and B5 samples show the presence of calcite which can act as fluxing agent in the ceramic body during firing. In Fig. 1 are plotted the spectra of two bentonites typologies (B2 and B4) which correspond to different rheological behaviours in suspensions state as reported in the rheological discussion.

The selected bentonites were added to the Sardinian clay in order to prepare Series I and II mixtures resulting from the substitution of the foreign clays into the reference porcelain ceramic body with the compositions previously reported in Table 1.

In whiteware processing, as in the most of the other technical processes involving concentrated suspensions, it is very important to control the fluidity and the stabilization of the slurries in order to obtain suspensions with suitable rheological properties. With this focus, rheological measures were performed by using a procedure that permits to measure the

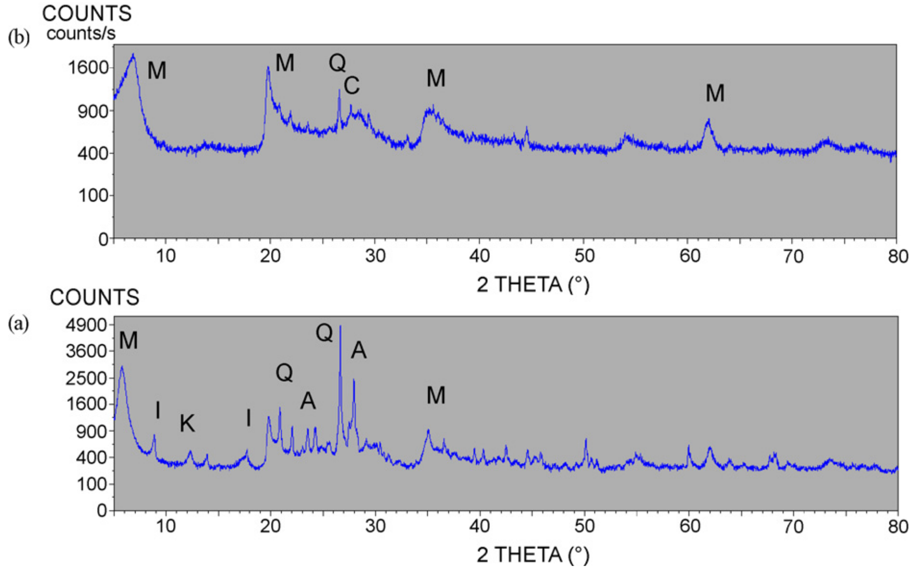

Fig. 1. XRD spectra of (a) B2 and (b) B4 bentonites samples. Q: quartz; M: montmorillonite; C: calcite; A: albite; K: kaolinite; I: illite.

complete flow curve (shear stress against shear rate) from which the yield stress and the apparent viscosity could be determined.

The flow curves of the two series highlight that the suspensions measured (solid content 66 wt%) are ‘‘non-Newtonian systems’’, and present a pseudo-plastic behaviour. The addition of plastic materials into the bodies increases the apparent viscosity as a function of the bentonite typology with respect to the standard one but for all the samples the yield stress is not present. While adding bentonite a reverse flow phenomenon is present due to the formation of a three-dimensional gel. The gel derives from the interaction of the electric double layer on the faces and edges of the clay particles [10]. All the flow curves exhibit a hysteresis cycle confirming the time dependence (thixotropy) of the suspensions. In Fig. 2 the curves obtained for the Series I mixtures compared with the reference one are reported. The viscosity data are considered at the maximum shear rate ($\dot{\gamma} = 200\,\mathrm{s}^{-1}$) in order to analyze the suspensions behaviour in critical steps such as milling, pumping, etc (Table 5). It is important to note that only for three samples (GB2, GB5, and UB5) the viscosity values are out of the workability range (200–350 mPa.s).

For the analysis of time dependence behaviour, on–off procedure was applied. This technique is useful to characterize the kinetics of the structural build-up processes which take place in a thixotropic system at rest conditions [11] (during stocking). Thixotropy is closely connected with the structural modification undergone by the disperse phase at shear rate constant with time. In fact the τ^* value (defined as the ratio between the stress overshoot and the stationary or quasi-stationary stress value attained before the shear interruption) increases with the rest time to reach values higher than 1, that is a typical feature of thixotropic system. Values close to 1 (τ^*) indicate a poorer tendency of the clay particles to rebuild networks. This characteristic is preferable in the industrial ceramic process. From the on–off tests the trend of τ^* with the rest time (t_R) was analyzed (Figs. 3 and 4). The τ^* vs. t_R has highlighted that the mixes of Series I and II prepared with B5

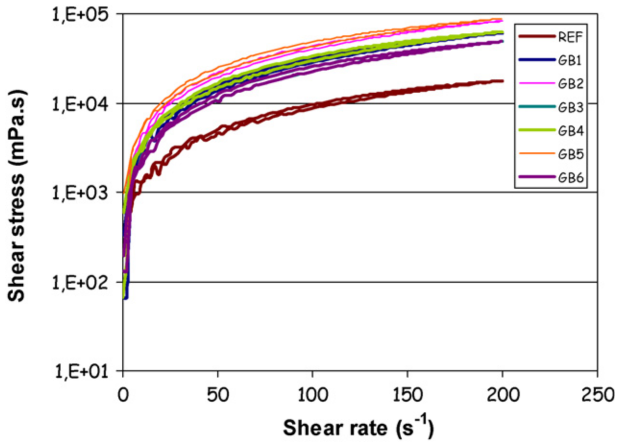

Fig. 2. Flow curves for the mixtures of Series I compared with the reference.

Table 5
Apparent viscosity values of the mixture suspensions measured at maximum shear rate (200 s^{-1})

MIX	Apparent viscosity (mPa . s)
REF	200
GB1	340
GB2	420
GB3	300
GB4	320
GB5	440
GB6	240
UB1	320
UB2	350
UB3	225
UB4	200
UB5	520
UB6	160

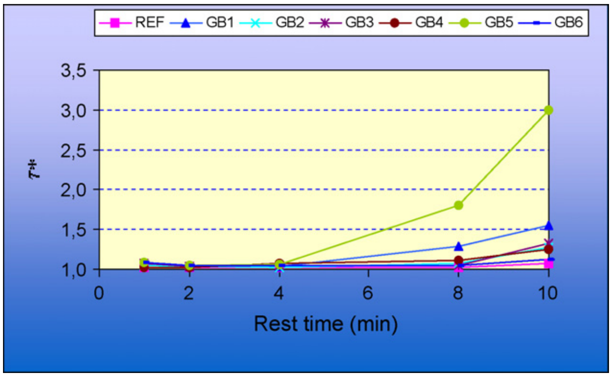

Fig. 3. Correlation of the experimental ratio τ^* vs. rest time for mixtures of Series I compared with the reference.

present the worst results (high thixotropy). Besides, all the mixes of Series II are less thixotropic than those belonging to Series I, that is due to need of a high rest time to build up.

Regarding the particle-size distribution no differences between the mixtures containing added clay with respect to the reference one were found. Besides, the residue measured is similar for all the samples. The suspensions prepared exhibit a bimodal particle-size distribution with two peaks (3.4 and 20 μm) and the D_{50} was about 8–9 μm (Fig. 5). On the basis of these considerations differences should not be found in pressing and sintering phases due to the particle size with the mixtures prepared.

After pressing the new formulation samples were submitted to mechanical tests in order to evaluate the effect of the different bentonites added. The breaking strength values measured in green are not better than the standard one, but in the dried samples emerged that the BS are higher than the standard in all the mixtures, confirming the effect of the montmorillonite (Table 6). It is interesting to note that the best data were obtained using B3, B4, B5, B6 for the Series I and B2, B4 and B5 for Series II.

Fired specimens were tested by LS% and WA% in order to verify the densification degree (Table 7). In general, it is possible to make some important remarks regarding the samples firing behaviour in comparison with the reference one: the GB1 and GB2 mixtures show LS% values and WA% values slightly higher probably as a consequence of the presence of kaolinite; GB6 mixture highlights a densification degree very similar to that of the reference one. In accordance with the industrial tolerance (±0.5) GB3, GB4, GB5 and GB6 gave satisfactory results. For all compositions of the Series I the WA% results were satisfactory with respect to the reference and the market requirements (<0.1%). On the other hand, Series II samples put out LS% values higher than the industrial tolerance. This phenomenon is due to the substitution of the illitic clay (U), having less shrinkage, with kaolinitic clay and bentonite, only UB5 can be accepted [12]. The LS% values of the second series indicate that the use of the trial clay mixtures can cause some calibration problems in the ceramic tile, although UB5 is a mixture most similar to the reference one. The WA% values were similar to the reference except to the mixtures UB1 and UB2 (the reason is the same referred for GB1 and GB2).

In the ceramic industry, the CIELab* method is the most utilized to determine the whiteness and the colour of the tiles by measuring the three parameters L^* (brightness), from absolute white $L = 100$ to absolute black $L = 0$, a^* (green–red) and b^* (blue–yellow) elaborated from the visible spectra [13]. The parameters data of the mixtures compared with the reference are collected in Table 7. For the Series I it is possible to note that the GB2 and GB3 mixtures show a L^* and a^* higher values than the standard one, while GB4, GB5 and GB6 are worst; GB1 is

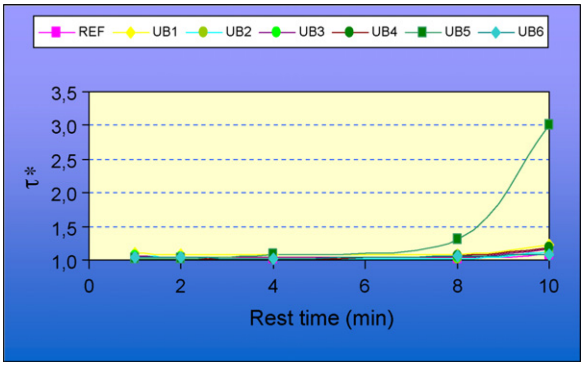

Fig. 4. Correlation of the experimental ratio τ^* vs. rest time for mixtures of Series II compared with the reference.

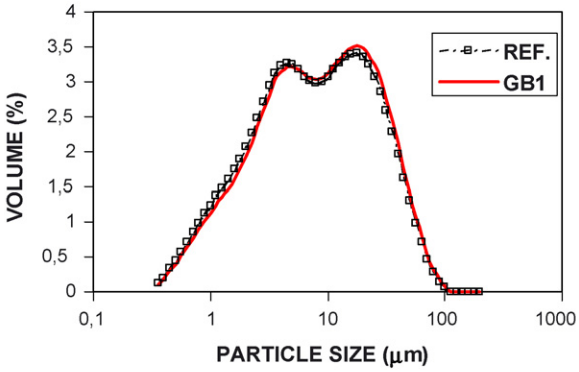

Fig. 5. Cumulative and distribution particle-size distribution curves for the reference body and GB1 mixture.

Table 6
Breaking strength (kgf cm^{-2}) for green (g) and dried (d) mixtures compared to the porcelain stoneware reference body

	BS (g)	BS (d)
REF	12.7	33.5
GB1	8.4	35.4
GB2	8.0	36.2
GB3	9.3	38.0
GB4	9.1	43.0
GB5	9.1	39.4
GB6	8.9	38.3
UB1	7.65	34.3
UB2	8.15	35.3
UB3	8.16	34.9
UB4	7.29	42.7
UB5	8.78	35.1
UB6	7.56	33.2

Table 7
Sintering (LS% and WA%) and colorimetric (L^*, a^*, b^*) parameters of the mixtures studied

	LS (%)	WA (%)	L^*	a^*	b^*
REF	6.31	0.04	65.91	1.71	11.30
GB1	7.24	0.07	65.51	2.56	10.43
GB2	7.19	0.06	66.03	2.46	10.46
GB3	6.81	0.03	66.52	2.33	10.21
GB4	6.84	0.05	64.04	2.64	10.37
GB5	6.56	0.07	64.13	2.47	10.12
GB6	6.55	0.06	64.48	2.78	10.46
UB1	7.25	0.23	67.15	2.52	10.50
UB2	7.18	0.34	67.81	2.48	10.24
UB3	7.16	0.09	66.32	2.44	10.21
UB4	7.11	0.06	65.23	2.39	10.35
UB5	6.88	0.07	66.08	2.49	10.21
UB6	7.44	0.06	57.48	2.69	7.91

similar to the reference one. For all samples the b^* values are lower than the reference one (less yellow). These effects could be caused by the presence of higher amounts of potassium feldspar in these compositions with respect to the reference. From the Series II emerged that L^* values obtained from UB1 and UB2 are higher than the reference product (porcelain stoneware) probably because of the lower fusibility (and so the lower oxidation of the chromophore elements oxides), while only UB6 shows the worst L^* value (as we saw for GB6) as a consequence of the different balance between chromophore elements (Fe and Ti) present into the bentonites added.

4. Conclusions

With the G clay (Series I) substitution, the mixtures obtained became rich in plastic components and this worsens the rheological behaviour, increasing the viscosity and the thixotropy. Nevertheless, some bodies (GB3, GB4, and GB6) were within the workability range (200–350 mPa.s^{-1}) and could be suitable for the preparation of commercial mixtures. Besides, they show breaking strength values on dried samples higher than the standard one. From the technological and colorimetric tests, the mixtures (GB3, GB4, GB5 and GB6) have shown values close to commercial product. These considerations permit to hypothesize the possibility to use the Italian clay, added with some typologies of bentonites in order to obtain porcelain stoneware bodies.

On the other hand the results of technological tests for the mixtures with the U clay substitution (Series II) show linear shrinkage higher than the reference especially for UB1 and UB6 mixtures; only the UB5 mixture is similar to the standard one (it shows a good BS in dried sample too) so its behaviour can be compared to that of the reference commercial product.

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