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Effect of coating on cleanability of glazed surfaces

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Abstract

The aim of this study was to establish the effects of different coatings on the cleanability of glazed ceramics. The surface properties were examined with a contact angle meter, a contact profilometer and a confocal microscope. The surfaces were soiled with three radiochemical model soils: inorganic particle soil, organic particle soil and oil soil. Soil adhesion on surfaces was measured with a quantitative radiochemical procedure. Generally, cleanability of the particles present in the model soil was found to be affected by the roughness of the surfaces; however, the cleanability of the oil in the model soils correlated with the contact angle of water on the surfaces. Coating of glazes, especially with fluoropolymer film, generally increased the contact angle values. The coatings affected the cleanability of ceramics somewhat: particle soils were removed most efficiently from glazes coated with TiO_2 and Zr. By contrast the oil soil residues of the fluoropolymer surfaces were the lowest. The cleanability results of the three model soils based on inorganic or organic particles or oil were different indicating differences between the cleanabilities of these main components of the soils.

Keywords: Surface; Cleanability; Chemical properties

1. Introduction

During recent years increasing effort has been directed to enhancing the cleanability of glazed surfaces with different types of additional surface coatings. Although some coatings are frequently used on, e.g. sanitary ware, very little information concerning the behaviour of the coatings in everyday life environments is available. The additional coatings used to render self-cleaning or easy-to-clean the surfaces should not change other properties such as surface appearance or roughness; rather they should provide the surface with an added new value, e.g. enhanced cleanability.

Kuisma et al.[1] examined the effects of surface topography of different compositions and surface coatings of glazed ceramic tiles on their cleanability. The results showed that there were clear differences in the soiling tendencies of glazed surfaces having different morphologies. It was generally concluded that the rougher the surface, the higher the amount of soil adhered to it. Soiling and cleaning degree of traditional glazed surfaces, consisting of different crystalline phases embedded in a glassy phase, have been reported to depend rather on surface micro- and macro-roughness than on their chemical composition.[2] The intensity of soiling of polished stoneware tiles has been found to be directly related to the values of R_a (surface roughness), P_{ro} (pore roundness) or P_{ma} (amount of macro-pores in the 1–50 μm range).[3] Smooth surfaces with round pores and with only a few coarse pores were the most soil resistant. Soil resistance and cleanability of white porcelain stoneware tiles have been reported to depend on the polishing process and on the microstructure of the surface.[4] Rough surfaces were easy to soil and hard to clean.

In general, the methods used to estimate cleanability of ceramics have been based on the variation of the colorimetric CIELAB parameters, caused by the soil attached to the surface, and measured by spectroscopic techniques. The surface colour offers an easy way to compare the cleanability of the surfaces, but

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does not necessarily correlate with the absolute amount of soil attached to the surface. Cleanability of resilient flooring materials has earlier been examined with radiochemical methods.[5,6] Radiochemistry has also been used to determine soil accumulation on plastic surfaces.[7] Measuring the amount of soil on the surface with radiochemical methods would provide quantitative informations useful to study different cleanability performances. Pesonen-Leinonen et al.[7] also reviewed the literature on these topics, but no published information concerning the use of radiochemical methods in cleanability studies of ceramic materials was available.

This paper is the first of a series, in which the properties of additional coatings on glazed ceramics are examined and discussed. The focus of this first paper is to compare the cleanabilities of different surfaces through radiochemical methods. The cleanabilities of inorganic and organic particle and oil soils are presented. The radiochemical determination of soiling tendency is able to quantify soil entrapped in surface flaws such as cracks or cavities, thus providing an accurate estimate of soil residues on surfaces. In the subsequent papers the influence of UV-radiation on cleanability of TiO_2-coated materials and the effect of chemical wear of the surface on its cleanability will be examined and discussed.

2. Experimental

2.1. Coating of glazed surfaces

The materials evaluated are presented in Table 1. Two matt (3A and M) and two glossy (K and S) glazes were used as substrates which underwent three different surface treatments. All glazes were commercial products with the only exception of 3A being an experimental laboratory-made glaze. The main crystalline phase in the commercial glazes was zircon, whereas diopside was found in the experimental one. A commercial fluoropolymer (F) and experimental sol–gel derived zirconia (Zr) and titania (Ti) films were used as surface coatings. The sanitary ware glaze (S) was coated only with the fluoropolymer film. Manufacturing of the surfaces has been presented earlier.[1] The firing times and temperatures are presented in Table 1. The glaze 3A was fired in a laboratory furnace, whereas the other glazes were fired in industrial kilns. The zirconia-coated samples were first heat-treated for 1 h at 300 °C, after which the temperature was increased to 600 °C ($\Delta T = 5$ °C/min) for maturation for 30 min. The titania-coated glazes were heat-treated for 55 min at 500 °C. The fluoropolymer-coating was applied at room temperature.

2.2. Determination of surface properties

Topography was measured with both a whitelight Confocal Microscopy (COM NanoFocus μSurf®) and a contact profilometer (KLA Tencor P15). The topography is represented by the three-dimensional roughness parameter S_a, which is the arithmetic mean of the absolute distances of the surface points from the mean plane. The value of S_a provides the roughness as well as spatial and hybrid information for three-dimensional surfaces (DIN EN ISO 4287).

When using COM, the roughness was measured with a cut-off wavelength of 250 μm and with a lens giving 20× magnification. The vertical resolution of the measurement was approximately 6 nm and six replicates were performed. With the contact profilometer three replicates of three-dimensional roughness profiles were measured. The vertical resolution of this profilometer is 1 nm and its horizontal resolution is 0.5 μm. 3D-scans were 500 μm in length; scan speed was 10 μm/s and sampling rate 20 Hz. The vertical resolution range was 327 μm and resolution was 1.9 nm. A Gaussian filter with short wavelength cut-off (off) and long wavelength cut-off (250 μm) was used to separate macro-roughness (waviness) from micro-roughness. Macro-roughness is defined as waviness and micro-roughness as roughness profile. The choice of parameters for profilometry has been presented earlier.[1,8] The R_a value, usually stated in micrometer units, is the most commonly used descriptor of surface roughness.

Table 1
Crystal phase composition, coating and firing temperature and time (modified from Kuisma et al.[1])

Code	Firing	Crystalline phases in glazes	Coating	Peak firing temperature/firing cycle
3A	Laboratory furnace	Diopside	None	1260 °C/7.5 h
3AF			F fluoropolymer	
3AZr			Zr zirconia (sol–gel)	
3ATi			Ti titania (sol–gel)	
K	Industrial kiln	Zircon	None	1215 °C/55 min
KF			F fluoropolymer	
KZr			Zr zirconia (sol–gel)	
KTi			Ti titania (sol–gel)	
M	Industrial kiln	Zircon	None	1215 °C/55 min
MF			F fluoropolymer	
MZr			Zr zirconia (sol–gel)	
MTi			Ti titania (sol–gel)	
S	Industrial kiln	Zircon	None	1215 °C/18 h
SF			F fluoropolymer	

In order to compare materials and measurement devices, water contact angle was also measured using two similar contact angle meters (type KSV CAM 100). With both instruments, static water contact angles on the experimental surfaces before soiling were measured. A water drop (ultrapure water Milli-Q) was placed on the surface and imaged for 20 s, collecting one image per second. Determination of contact angle was based on the Young–Laplace equation. The result was the mean of the drop on ten replicate samples with instrument 1 and the mean of the drop on five replicate samples with instrument 2.

2.3. Soiling and cleaning of the ceramic materials

Cleanability was studied using three different model soils containing typical components of the environments where ceramic materials are used, such as floors and walls in public buildings and houses, especially in bathrooms. The model soil 1 contained chromium oxide Cr_2O_3 as inorganic particle, whereas model soil 2 contained organic particle chromium acetyl acetonate, $C_{15}H_{21}CrO_6$. Model soil 3 contained a triglyceride (triolein, $C_{57}H_{104}O_6$) representing a model of natural oils and sebum. The particles of model soils 1 and 2 were labeled with the gamma-ray emitter ^{51}Cr and the oil soil with the beta-ray emitter ^{14}C (Table 2). The prerequisite for selection of the isotope is that it is chemically bound to the soiling agent. In this study chromium compounds were irradiated in order to obtain radioactive ^{51}Cr-isotope. The triolein contained ^{14}C-isotope. The cleanabilities of different components of model soils were estimated by measuring the amounts of different radio-isotopes on the surfaces.

All the surfaces mentioned in Table 1, including surfaces before and after the surface treatments, were subjected to one soiling and one cleaning cycle. Soiling was carried out with the procedure described in detail by Pesonen-Leinonen et al.[7] The soil was applied as a liquid suspension (50 μl) on the middle of the sample with a pipette and 1-propanol was used as a carrier to assist dosage (Table 2). The soil was left to dry for 24 ± 2 h at room temperature. Cleaning was carried out with a Mini Cleanability Tester as described earlier.[7] The cleaning head was equipped with a microfibre cloth (Freudenberg Household Products Oy Ab).[7,9,10] The estimated pressure applied to the sample was 25 kPa, velocity 30 rpm and the number of revolutions was three. The material of the microfibre cloth was polyester (100%) and the pile length was 8 mm. The cloth was moistened at 100% moisture regain with 5% detergent solution. The detergent was a weakly alkaline model detergent which contained triethanol amine soap of fatty acids (1.75 wt%), non-ionic surfactant (C13-oxoalcohol ethoxylate, 9 wt%) and tetrapotassium pyrophosphate (5 wt%) (Farmos).[7,8,10]

2.4. Measurement of cleanability using the radiochemical method

Two different methods, a gammaspectrometric method and a liquid scintillation counting, were used for the evaluation of surface cleanability. In both methods, the radioactivity of the surface was compared to the amount of the labeled component of soil on the sample. The cleaning result was calculated as the proportion of the labeled component of soil after cleaning compared to that after soiling. Five replicate tests were performed for each test combination. The radiochemical method also detects soil which has penetrated into surface flaws such as cracks or cavities in the surfaces, thus giving the total amount of soil attached to the material.

The cleanabilities of the model soils labeled with the gamma-ray emitter ^{51}Cr (Table 2) were determined by a gammaspectrometric method using an NaI(Tl)-scintillation crystal described in detail by Määttä et al.[10] The counting system comprised of a 2 in. × 2 in. NaI(Tl)-crystal detector (Bicron Corporation, Ohio, USA) coupled with a multichannel analyser and standard electronics (Canberra Inc., Connecticut, USA). The number of counts was recorded from 2 min to 5 min depending of the activity of the sample. The radioactivities of the soiled samples were measured before and after cleaning. The results were calculated by subtracting the activity of the background and correcting the results for radioactive decay.

The cleanability of model soil labeled with the beta-ray emitter ^{14}C (Table 2) was measured using liquid scintillation counting. Due to the hardness of ceramic tiles, the samples could not be cut into small pieces needed for direct measurement of the amount of soil left on the surfaces after the cleaning step without the risk of uncontrolled reduction of activity. Therefore, the activities of the mop cloths after cleaning were measured instead of the surfaces. The amount of soil was then calculated from the results of activities of mop cloths and of soiled surfaces. The mop cloths were oxidized in an oxidizer and the radioactivity was measured using liquid scintillation counting. The counting system consisted of a scintillation counter (Wallac 1411 Liquid Scintillation Counter) and a measuring program (1414 WinSpectral^{TM}). The measurement time was 10 min. Calculation of the results included the attenuation equalizer and subtraction of the background. Correction of radioactive decay was not needed because of the long half-life of carbon.

Table 2
Compositions and amounts of model soils used in the radiochemical study

Type of the model soil	Components of the model soil				Amount of soil (μl) on a disc
	Chromium compound ($m = 0.40$ g)	Solvent ($V = 10.0$ ml)	Fatty acid ($V = 0.60$ ml)	Radioisotope	
1. Inorganic particle soil	Chromium(III) oxide (Cr_2O_3)	1-Propanol	Triolein ($C_{57}H_{104}O_6$)	^{51}Cr	50
2. Organic particle soil	Chromium acetyl acetonate ($C_{15}H_{21}CrO_6$)	1-Propanol	Triolein ($C_{57}H_{104}O_6$)	^{51}Cr	50
3. Oil soil	Chromium(III) oxide (Cr_2O_3)	1-Propanol	Triolein ($C_{57}H_{104}O_6$)	^{14}C	50

In all soils, triolein refers to glyceryl trioleate.

2.5. Statistical analysis

The cleaning result was calculated as the ratio between of the soil residue after cleaning and the amount of soil on the surface after soiling. Statistical analyses were performed using SPSS version 12.0 (SPSS Inc., Chicago, IL, USA), based on the mean values of the results. The cleanability results for each of the three soils were analysed separately. Analysis of variance was used to examine differences between the materials and treatments. Bivariate correlation analysis (Pearson's correlation coefficients, two-tailed test of significance) was used to examine the possible correlation between roughness, contact angles and soil residue. The significance used was 0.05 in analysis of variance and 0.01 in analysis of correlation.

3. Results

3.1. Surface properties

Surface topography was described with two different instruments. The S_a roughness values obtained with the profilometer were in general higher than those given by COM. However, both methods gave the same trend for roughness. The difference in the roughness was assumed be due to the higher resolution of nanoscaled surface variations with the profilometer or different surface areas used for the measurements. Significant correlations between the examined roughness parameters were observed (Pearson's correlation coefficient $r = 0.929$).

The roughness parameters S_a are given in Table 3. Values of the parameter S_a varied between 0.04 μm and 0.58 μm measured with COM. The glaze 3AF had the highest roughness values, whereas the lowest values were measured for glaze S. The contact profilometer gave roughness values of a similar order to those of COM. The S_a values given by the contact profilometer varied between 0.11 μm measured for glaze S to 0.73 μm measured for glaze 3A (Table 3). There were no statistically significant differences between roughness of the uncoated and coated glazes ($p > 0.05$). This implies that the coatings can be used to modify glazed surfaces without affecting the desired surface roughness.

The contact angle of ceramic surfaces is usually reported to vary in the range of 30–50°. This variation in contact angle values is a result of different compositions of the measured surfaces and different measurement methods or equipment. In this study surfaces with the same composition were measured with one method but with two different instruments. The contact angles of water on glazes are presented in Table 3. The contact angle values measured with the first instrument varied between 31° and 97° depending generally on the coating. The contact angle values examined with the second instrument were somewhat lower, varying between 28° and 82°. The Pearson's correlation coefficient r was 0.679 between contact angle values for both measurement methods. However, when the results of the two methods were compared with each other, the contact angle was the same only for one of the fourteen measured surfaces, namely glaze M (Table 3). Furthermore, when the materials were put in order from the lowest contact angle to the highest one, no general similarity was obtained with the two methods.

The contact angles of the surface SF was the highest in both methods and that of the glaze M (instrument 1) or K (instrument 2) the lowest. However, the magnitude of the contact angles was the same for all the surfaces, i.e. ceramic surfaces had values between 30 and 50 for the uncoated samples using both methods. Slightly increased contact angles were measured for the zirconia-coating. The contact angle for the titania-coatings was of the same order as for the substrate glaze surface. However, titania is known to decrease the contact angle when interacting with light. The experimental surfaces were not exposed to any special UV-light treatment, but neither were they covered to avoid the influence of illumination during the experiments. These phenomena will be examined in a later study. Both contact angle measurements indicated that the contact angle of the fluoropolymer-coating was increased to values close to or within the range of hydrophobicity. Thus, the coating is likely to impact an easy-to-clean or self-cleaning effect to the surface. The self-cleaning effect depends on oxidizing mechanism due to the interaction with light.

3.2. Cleanability of the surface

The quantitative radiochemical determination method used for examining cleanability of surfaces provided detailed information on the attachment of different soil components to the surfaces (Fig. 1). The inorganic particle soil (model soil 1, labeled with ^{51}Cr), containing chromium oxide and triolein, was generally removed more efficiently from almost all surfaces than the two other model soils. However, the additional coatings did not statistically significantly affect the cleanability of the inorganic particle soil ($p > 0.05$). The morphology of the substrate glaze was found to affect the cleanability ($p = 0.003$). The inorganic particle soil (model soil 1) was removed most efficiently from the rough surfaces, i.e. glazes 3A and M (Figs. 1 and 2).

The soil used to describe the attachment of organic particle soil (model soil 2) contained chromium acetyl acetonate (labeled with ^{51}Cr) and triolein. In this case the coating affected the cleanability ($p = 0.003$). Fluoropolymer-coating increased soil attachment to the surface, whereas zirconia somewhat decreased the amount of soil left on the surfaces after cleaning. The glaze

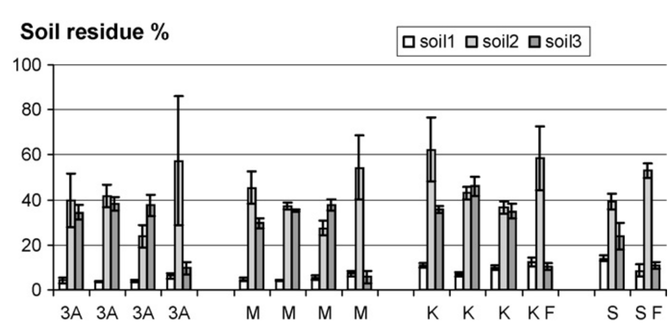

Fig. 1. Effects of coating on the cleanability of ceramic materials using different ^{14}C and ^{51}Cr labeled model soils, explained in detail in Table 2. Column: mean of five replicates, bar: standard error (±S.E.).

Table 3
Contact angles and roughness parameters presented as means of measurements ± standard errors of means (±S.E.)

Sample code[a]	Contact angle		Roughness parameter (μm)	
	Instrument 1	Instrument 2	S_a (profilometer)	S_a (confocal microscope)
3A	33 ± 2	41 ± 2	0.73 ± 0.1	0.54 ± 0.004
3AF	92 ± 2	68 ± 9	0.56 ± 0.04	0.58 ± 0.004
3ATi	45 ± 2	32 ± 2	0.69 ± 0.01	0.38 ± 0.02
3AZr	64 ± 3	32 ± 4	0.65 ± 0.03	0.52 ± 0.004
K	32 ± 0.7	28 ± 1	0.22 ± 0.04	0.09 ± 0.02
KF	95 ± 2	81 ± 2	0.25 ± 0.05	0.12 ± 0.001
KTi	34 ± 0.8	29 ± 7	0.28 ± 0.06	0.09 ± 0.004
KZr	37 ± 0.6	50 ± 2	0.22 ± 0.03	0.08 ± 0.003
M	31 ± 1	31 ± 2	0.69 ± 0.7	0.44 ± 0.004
MF	92 ± 2	79 ± 3	0.61 ± 0.05	0.41 ± 0.01
MTi	45 ± 0.8	43 ± 7	0.63 ± 0.09	0.38 ± 0.01
MZr	61 ± 2	46 ± 5	0.61 ± 0.03	0.36 ± 0.01
S	34 ± 2	38 ± 6	0.11 ± 0.03	0.04 ± 0.001
SF	97 ± 2	82 ± 5	0.16 ± 0.04	0.07 ± 0.001

[a] F refers to fluoropolymer, Ti to titanium and Zr to zirconium coating (codes are presented in Table 1).

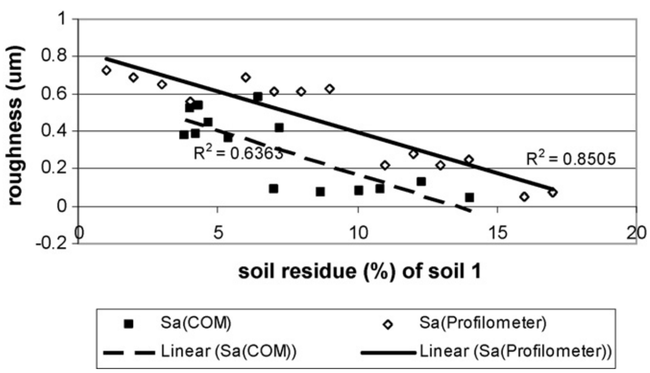

Fig. 2. Correlation between the roughness parameters and cleanability of model soil 1, calculated from means of the results.

material was observed to affect surface topography (Table 3), but did not statistically significantly affect the amount of the organic particle soil residue ($p = 0.675$). Thus, the surface morphology of the glaze was not found to influence the cleanability of soil typical for organic particles in the everyday environment.

The model soil 3 comprised oil soil containing inorganic particles (Cr_2O_3) and triolein, the latter being labeled with ^{14}C. The oil soil was found to be removed from the surfaces very differently depending on the coating ($p = 0.000$). In contrast to the particle soils the soil residues of oil soil on the fluoropolymer surfaces were the lowest. As in the case of organic particle soil, the glaze material or topography had no statistically significant effect on the cleanability ($p = 0.684$) of oil soil. However, Fig. 1 indicates that residues of the oil soil were the lowest on M surfaces. There was a significant correlation between contact angles and soil residue (Pearson's correlation coefficients $r = -0.816$ and $r = -0.739$ for soil residues and contact angles (instruments 1 and 2, respectively)) (Fig. 3).

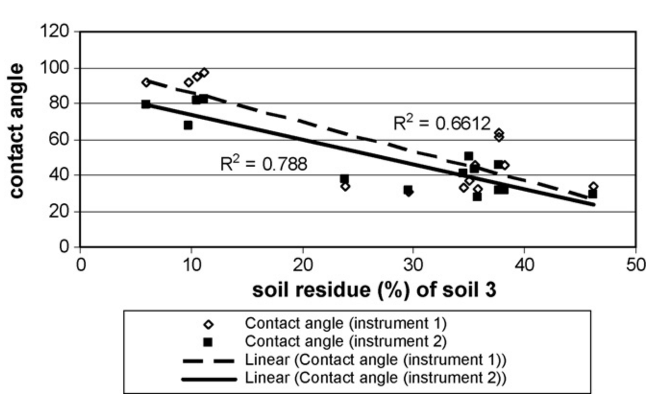

Fig. 3. Correlation between the contact angle values and cleanability of model soil 3, calculated from means of the results.

4. Discussion

Cleanability of the surfaces from particle components of model soils was found in this study to depend on the surface microstructure. Rough ceramic surfaces have been reported to give lower soil resistance and cleanability than smooth ceramics.[1–4] In this study model soil 1, representing inorganic particle contamination, was removed more efficiently from 3A and M surfaces, which were rougher than K and S surfaces. However, despite differences in the roughness parameters, all examined surfaces can be considered as rather smooth and therefore the effect of surface coarseness on cleanability was not clear. In the hygienic surface criterion for roughness is set at a maximum R_a value of 0.8 μm.[11] The roughness values of the surfaces studied were lower than 0.8 μm, thus indicating that their cleanability from the hygienic point of view should not be seriously affected by their maximum roughness level.

The surface microstructure had a general correlation with cleanability of the glazed surfaces from inorganic particle soil containing chromium oxide and triolein. In the case of oil soil containing ^{14}C labeled triolein and chromium oxide there was a significant correlation between contact angle and soil residue. According to the present cleaning results, it appears that surface microstructure affects the cleanability of particle components of soils and that surface chemistry affects the cleanability of oil components. This could be due to the different forms of the labeled components of the soils used. The particle components were in solid form and may only have attached to peaks of the surface structure.

The cleanability of the oil component could be explained by the hydrophobic or hydrophilic properties of the surfaces as described by contact angles. The fluoropolymer-coated ceramics were the most hydrophobic materials and had the lowest soil residues of model soil 3.

The radiochemical method provides more detailed information on cleanability than, e.g. a more commonly used semi-quantitative method, colorimetry. The radiochemical method also detects soil which has penetrated into microcracks in the surfaces, whereas the colorimetric method only detects soil interfering with the colour of the surface. When using the radiochemical measuring method it is evident that selection of the element to be labeled is critical for the final cleanability results. The cleanabilities of different components of typical soils can be measured using different radio isotopes. ^{51}Cr isotope can be used to label particles typical for inorganic and organic soils and a ^{14}C isotope to label oil soils. Due to the two different radioactive emitters used to label the different soils types, the interaction of the surface with different soil types in typical everyday life environments could be expressed.

In this study cleanability was determined on horizontal surfaces. Thus, the results can be applied to estimate the soil attachment and cleanability on different types of surfaces rather than to correlate with self-cleaning properties. Although active self-cleaning might decrease the effort needed for cleaning, horizontal indoor surfaces particularly require active cleaning in exception to vertical surfaces.

5. Conclusions

The radioisotope technique developed earlier to quantify the cleanability of plastic materials proved also to be suitable for glazed surfaces. The overall surface roughness varied depending on the phase composition of the glaze, whereas the additional surface films had only a minor effect on the roughness. Thus, glazed surfaces can be coated with additional films without changing the original roughness. The sol–gel derived titania- and zirconia-coatings had only minor influence on the contact angle of water. However, UV-light would be necessary for the titania-coating in practice, and the present measurements were performed without exposure to UV-light. These experiments will be presented in our subsequent paper. By contrast, fluoropolymer was found to increase the average contact angle of the surface close to the hydrophobic range.

It was observed that in the cases of particle model soils (soils 1 and 2) the surface structure (topography) affected the cleanability of the ceramic materials, but in the case of oil soil (model soil 3) the surface chemistry (indicated by contact angle) affected the cleanability. This indicates that the attachment of particles depended on the surface roughness but that the contact angle was important for the attachment of oils. The inorganic particle soil was removed more efficiently from surfaces than organic particle soil and oil soil, except in the case of fluoropolymer-coated glazes, from which the organic particle soil was removed less efficiently than oil soil. The coatings affected the cleanability of ceramics to some extent: particle soils were removed most efficiently from sol–gel (TiO_2 and Zr) coated glazes. By contrast, in the case oil soil, the soil residues of the fluoropolymer surfaces were the lowest. The cleanability results of different model soils were different indicating that there are differences in the cleanabilities of different components of soil. Different model soils and methods are therefore needed to determine surface cleanability.

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