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Enhancing Hygiene and Technical Properties of Ceramic Tiles through Moroccan Phosphate Additives

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Abstract

This study offers a simple solution to manufacture ceramic tiles with good technical and hygienic properties. This solution consists of integrating the phosphate product from Morocco in the industrial formulation of ceramic tiles, to produce materials in compliance with the criterion of the ISO standard. For phosphate products, three grades were studied: HG-high grade, MG-medium grade, and LG-low grade. The results show that for materials containing these additives, there is both an effect of the concentration and type of additive on technical and hygienic properties. The mechanical property is improved when the incorporated component was richer in P2O5. Only the use of HG-high grade and MG-medium grade as an additive at least 15% makes it possible to satisfy the mechanical requirement. The anti-biofilm effect of natural phosphate (PN) as an additive to manufacture ceramic tiles is confirmed, it may prevent bio adhesion and biofilm formation at almost 75% for the HG-high grade and MG-medium grade. This solution could interest professionals and all users who care about the state of hygiene of their ceramic materials sensible to the formation of biofilm, like orthopaedic implants, swimming pool tile…etc.

Keywords: Mining; Phosphate; Ceramic tiles; Bacterial bioadhesion; Biofilm; Mechanical properties

1. Introduction

Morocco's mining heritage is very rich in phosphates; it has been reported that it contains three quarters of the world's reserves, which allows it to rise among the four main universal producers and exporters (Hakkou et al., 2008; Hakkou et al., 2009; El Berkaoui et al., 2021). Moreover, around ten other minerals (zinc, lead, anthracite, iron, copper, barite, antimony, fluorite, cobalt, silver, and manganese) are regularly produced, and make it possible to enrich this heritage (Mehahad and Bounar, 2020; Zine et al., 2020). In addition, clays from Morocco occupy a predominant place in all sedimentary rocks. Their mineralogical and physicochemical properties arouse particular interest in many applications including water treatment, paint, barrier for pollutants, adsorbents, catalysts, and the manufacture of construction products (Harti et al., 2007). However, Morocco is known to be a major consumer of clay. The reserves of these materials in Morocco are largely sufficient to ensure totally or partially the supply of the ceramic industry (Harti et al., 2007; El Ouahabi et al., 2014). Ceramics products for interior, exterior, and wall coverings, for floors, for bathrooms, public hammams, and for ceramic swimming pools are among the sectors that constitute Morocco's cultural heritage. With development and urbanization, their productions are increasingly in demand and modernized to meet all the requirements, aesthetics, thermal insulation, energy saving, durability, etc. Unfortunately, their productions in the field of ceramics remain most often artisanal and semi-industrial (dominated by family companies and artisans) and many problems remain and encounter daily by ceramists to bring their products into conformity with local and global technical and regulatory requirements. Despite the abundant literature on this subject (Harti et al., 2007, Sadik et al., 2012), the scarcity of specific studies on the raw material used and the used formulation are often responsible for many problems during production, which we can cite breakage, deformations or malformations of finished products. These problems significantly limit yields. Controlling the quality of ceramic tiles requires very broad knowledge in various scientific fields (geology, mineralogy, geochemistry, formulation, thermodynamics, mechanics, etc.) because of the multitude and diversity of steps in the manufacturing process. In a context of hygiene, given that ceramic coatings have in most cases faced with a humid environment, this makes them conducive to the formation of biofilms (Boutaleb et al., 2008a,b). This work aims to propose a simple solution, which consists in combining local mining resources in Morocco to produce ceramic products of good technical and hygienic quality, in compliance with ISO standards, and thus avoid the enormous economic losses associated with their production in industries that are currently finding it difficult to produce a compliant material made 100% from local clays. The effect antibiofilm of the natural phosphate (PN) as additives has already been proven in previous research work, in particular on the biomaterials of orthopaedic implants based on calcium phosphates and in particular hydroxyapatite (Ferraz et al., 2004; Ramos et al., 2018). This effect will be confirmed also during this research work for ceramic materials by using various grades of phosphate: HG-high grade, MG-medium grade, and LG-low grade incorporated into the base formula used to produce the ceramic tile products.

2. Material and method

2.1. Materials and characterisation

The phosphates used are from the Cherifien Phosphates Office Group (OCP) – Ouled Abdoun - Beni-Idir (Khouribga in Morocco). Several grades are used: HG-high grade, MG-medium grade, and LG-low grade. Their content in BPL and P_2O_5 are shown in Table 1.

Table 1. Percentage in BPL and P_2O_5 in different products

	BPL* (%)	**P_2O_5 (%)**
High grade (HG)	> 69.5	31.80
Medium grade (MG)	68<BPL<69.5	$31.12<P_2O_5<31.80$
Low grade (LG)	61<BPL≤68.0	$27.91<P_2O_5<31.12$

*Phosphate concentration is usually expressed as a percentage of P2O5 or its $Ca_3(PO_4)_2$ equivalent, known by the acronyms B.P.L. (Bone Phosphate of Lime): P_2O_5 = BPL x 0.4576.

The abundant local clay from the region of El Gara (33°16' 42.7"N 7°13' 39.6" W) was used to prepare the studied mixtures. This region is part of the Triassic basin of Mohammedia– Benslimane-El Gara-Berrechid (Afenzar and Essamoud, 2017). Particle size analysis of clays is carried out according to the method described by the United States Department of Agriculture (USDA) (ASTM, 1972). The chemical composition of all geomaterials used was determined by X-ray fluorescence using the WDXRF, S4 Pioneer device supplied by BRUKER S8® (Boutaleb et al., 2020a,b). The chemical structure was studied by infrared spectroscopy using Affinity-1S SHIMADZU spectrometer, in the range 400–4000 cm^{-1} at a resolution of 16 cm^{-1}.

2.2. Manufacturing of the ceramic tiles' specimen

For the preparation of the tile specimens, the various mixtures were introduced in a dry state and homogenized in a ball mill jar of 500 g capacity. The mixture is added to distilled water (4%) and Fluicer® deflocculates (1%). Mixing of all the ingredients lasts 30 min. The slurry recovered is dried in an industrial roller dryer at a temperature of up to 110 ° C for 30 min. The product obtained is then ground, sieved (particle size <63 µm) and humidified using a sprayer within limits of 4 to 6% water. Pellets approximately 60 mm in diameter and 7 mm thick were prepared by manual pressing under uniaxial pressure (200 bars) using a hydraulic press of the Sassuolo Lab ® type. A final firing step at 1200 °C is applied, which lasts 40 min (Boutaleb et al. 2020a,b,c). The tests from F1 to F11 were organized according to Table 2.

Table 2. The different formulas studied Technical and anti-bioadhesive properties

Formulas	**Clay**	**Additive**		
		LT	**MT**	**HT**
F1 (Control)	**100%**	0%	0%	0%
F3	95%	**5%**	0%	0%
F4	85%	**15%**	0%	0%
F5	70%	**30%**	0%	0%
F6	95%	0%	**5%**	0%
F7	85%	0%	**15%**	0%
F8	70%	0%	**30%**	0%
F9	95%	0%	0%	**5%**
F10	85%	0%	0%	**15%**
F11	70%	0%	0%	**30%**

F1, which does not contain a phosphate additive, consists of a test control and will make it possible to study the effect of the integration of the phosphate products. For each additive, the three percentages 5, 15, and 30% are tested.

2.3. Technical quality control of specimens

Mechanical properties of studied specimens were determined using Shimadzu® single-column machine, EZ LX series according to international standards NM ISO 10545-3 (2017), and NM ISO 10545-4 (2017). The firing shrinkage and the water absorption are determined using method described by Boutaleb *et al.*, (2020a,b,c).

2.5. Anti-bioadhesive properties of tiles

Adhesion tests were performed using bacteria indicating hygiene *Pseudomonas aeruginosa* (ATCC27853) and *Escherichia coli* (ATCC25922). For each bacterium, suspension with an optical density at 405 nm of between 0.7 and 0.8 is prepared according to the protocol described by El Omari *et al.* (2017, 2018). The adhesion test is conducted by immersing the surfaces in the bacterial suspension for 2 hours at 37°C (Boutaleb et al., 2008a,b; El Omari et al., 2017; El Omari et al., 2018). The surfaces are then collected, washed very gently with sterile distilled water held to remove any cells that are not adhered to and that may distort the count. A microscopic observation in Scanning Electron Microscopy (SEM) Philips, Model XL30 was also conducted for the determination of CFU per unit area and to study the anti-bioadhesive properties of tiles (Boutaleb et al., 2008a,b; El Omari et al., 2017; El Omari et al., 2018). Matlab Software was used to convert images of Scanning Electron Microscopy SEM to their digital form and then calculate the percentage of surface occupation by bacteria.

3. Results and Interpretation

3.1. Chemical and physical characterization of clays

The abundant clay from the El Gara region of Morocco shows a very high percentage of Silica and Aluminum (Table 3), this indicates, the presence of Kaolinite ($Al_2Si_2O_5(OH)_4$) which help to have low fire shrinkage and allows the manufacture of refractory materials (El Yakoubi et al., 2006; Laibi et al., 2017). The Alumina/Silica ratio provides information on the material's moisture permeability, the greater this ratio the greater the permeability (Sadik et al., 2012).

Table 3. Chemical compositions by X-ray fluorescence (wt %) and Particle size characterization (%) of used clay

Particle size characterization (%)	
Clay < 2µm	60
Sand 50-2000µm	15
Silt 2-50 µm	25
Chemical Characterization (%)	
SiO_2	55.700
Al_2O_3	17.000
Fe_2O_3	6.250
CaO	3.950
MgO	2.700
K_2O	4.550
Na_2O	0.400
P_2O_5	0.100

SO_3	0.130
TiO_2	0.800
MnO	0.150
BaO	0.055
Al_2O_3/SiO_2	0.305
Fire loss	7.035

The high amount of calcium (CaO) indicates the richness of the clays in calcite ($CaCO_3$) that help in acid-base environment stabilizing and help to generate of refractory monolith essential for thermal resistance. The overall composition of the other oxides (Fe_2O_3, MgO, K_2O, and SO_3) reaches a percentage of 9% according to the results of the chemical composition by X fluorescence (Table 3) which shows that our clay is not pure (El Yakoubi et al., 2006; Harti et al., 2007; Hakkou et al., 2016; Laibi et al., 2017). SO_3 when present improves plasticity (El Yakoubi et al., 2006; Laibi et al., 2017). The particle size analysis in Table 3 shows that the clay used is among the category of fine clays according to the conventional classification of the USDA (ASTM, 1972). This type of soil is characterized by high plasticity when humid or compactness when is dry.

3.2. Spectroscopic Data

The vibrations at 532 and 470 cm^{-1} shown in Fig. 1a, are attributable to the deformations of the Si-O-Al bonds and respectively where Al is hexacoordinate and Si-O-Si in the kaolinite. The bands at 702 cm^{-1} correspond to a vibration of the Si-O-Al bond where Al is tetracoordinate in kaolinite. Table 4 shows the wavenumbers with their corresponding functional groups as reported earlier by Sadik *et al.*, (2012). The results of this analysis are in accordance with the chemical analysis by X-ray fluorescence, indicating the presence of Kaolinite in the analysed material. The FTIR spectral data of different grades of natural phosphate (NP) are presented in Fig. 1b.

Table 4. Vibrational band for used clay and their attributions as reported by Sadik *et al.*, (2012)

Wavenumber (cm^{-1})	Attribution
Vibration at 908	Deformation of the Al-OH bond in kaolinite
Intense band centered around 1027 and the band at 640	Vibrations of elongations of the Si-O-Si bond in kaolinite in clay minerals
Bands at 3688 and 3619	Vibration of the O-H bond of the hydroxyl groups of kaolinite
Band at 1113	Vibration of the Si-O bond of kaolinite
Band at 1628	Deformation vibrations of the OH^- and H_2O groups

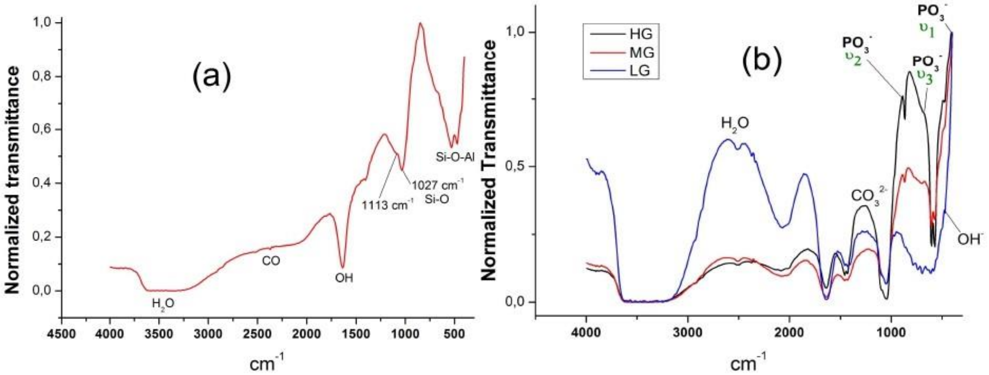

Figure 1. Fourier transform infrared spectrum of (a) clay used (b) different grades of natural phosphate (PN)

Different absorption bands were shown, indicating the presence of different functional groups on the NP surface. The intense band at 1040 cm^{-1} is attributed to the PO_3^{4-} group, corresponding to the anti-symmetric valence vibration band domain of the P-O bond. The doublet observed at 567 and 605 cm^{-1}, are attributed to the deformation vibration mode of the P-O bond. The band at 1095 cm-1 indicates the presence of HP_2O_4 groups. The absorption bands at 870 and 1450 cm^{-1} indicate the presence of CO_2^{3-} ions. The intense mid band around 1630 cm^{-1} is attributed to H_2O and CO_2 groups. The weak band at 3540 cm^{-1} indicates the presence of hydroxyl groups of the Ca(OH) group. A broad absorption band around 3700-3000 cm^{-1} indicates the presence of water molecules and/or hydroxyl groups (Mabroum et al., 2020)

3.3. Mechanical and technical properties of the specimens

According to Fig. 2, we observed that the gradual addition of the phosphate-rich additive helps to reduce weight loss with a more or less stable loss in diameter. This is less visible when the additive is LG-low grade probably because of the presence of impurities. The loss of weight often causes the formation of cavities and a reduction in density (Weng et al., 2003).

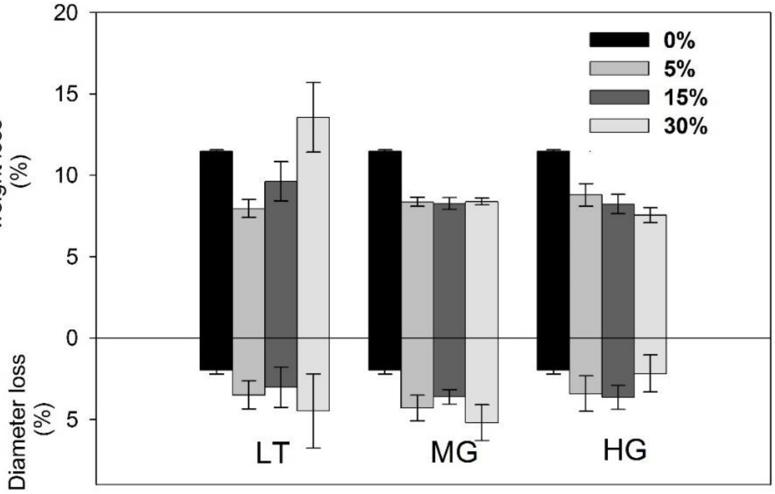

Figure 2. Evaluation of the weight and diameter loss in all the produced materials.

According to the standards applied in this field (NM ISO 10545-3, 2017; NM ISO 10545-4, 2017), Group III are materials characterized by water absorption percentage that exceeds 10%, and they are not necessarily the first grade. Group II are materials characterized by water absorption percentages between 3% and 10%. Group I are materials characterized by water absorption percentage less than 3%. For all these categories a minimum breaking force of 600 N (for a thickness <7.5 mm) is required (Boutaleb et al., 2020a,c).

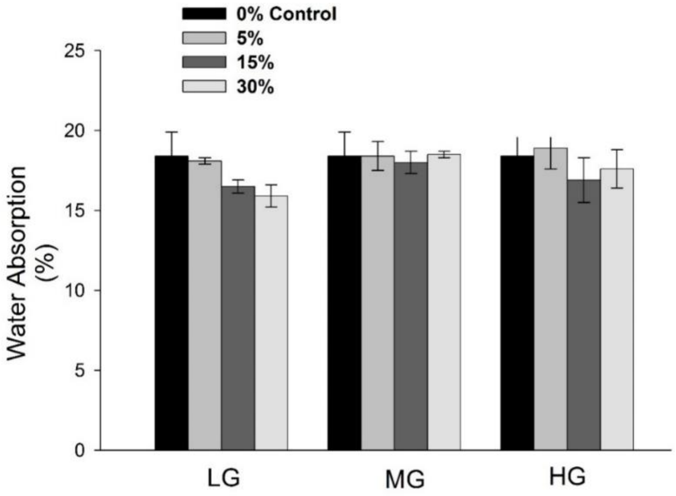

Figure 3. Evaluation of the water absorption in all produced materials.

Fig. 3 shows that for all materials, the integration of phosphate product additive does not significantly influence water absorption. When additive is HG-high grade and MG-medium grade the use of at least 15% of each one is necessary to satisfy the requirement on the mechanical properties. However, when additive is LG-low grade it is not possible to produce tiles that satisfy the mechanical requirements (Fig. 4).

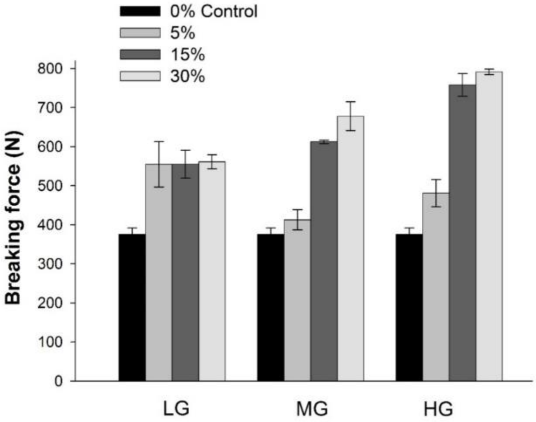

Figure 4. Mechanical properties of the tiles produced.

3.4. Studies of Anti bio-adhesion effect

According to Fig. 5 the adhesion score seems to depend both on the nature of the suspension of bacterial strains, the type of grade used, and the percentage integrated into the formulation of each grade. *P. aeruginosa* scores a higher adhesion score than *E. coli* in all the specimens studied. The adhesion score generally respects the following order: LG-low grade>MG- medium grade>HG-high grade and decrease when percentage of each additive is greater it is so positively correlated with the P_2O_5 richness. The richer the additive is by this element, the more the percentage of adhesion decreases. *P. aeruginosa* and for *E. coli*, are recorded respectively, a reduction of approximately 61% and 75% for HG-high grade as best results of adhesion inhibition.

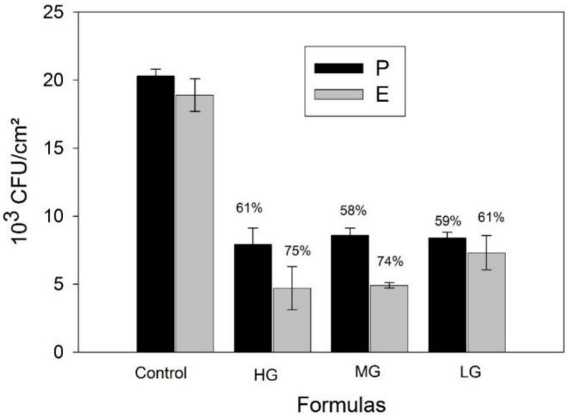

Figure 5. Bacterial count results after adhesion test of *P. aeruginosa* (P) and *E. coli* (E) on the studied specimens.

Fig. 6 show examples of SEM observations. Fig. 6 shows the appearance of the biofilm in the most colonized zones of the surface as a function of bioactive additive richness. It clearly manifested that a more complex and developed structuring of the biofilm is visible for low concentrations of bioactive additives, which appears more covered by probably the exopolymer matrix (slime) (El Omari et al., 2017; El Omari et al., 2018).

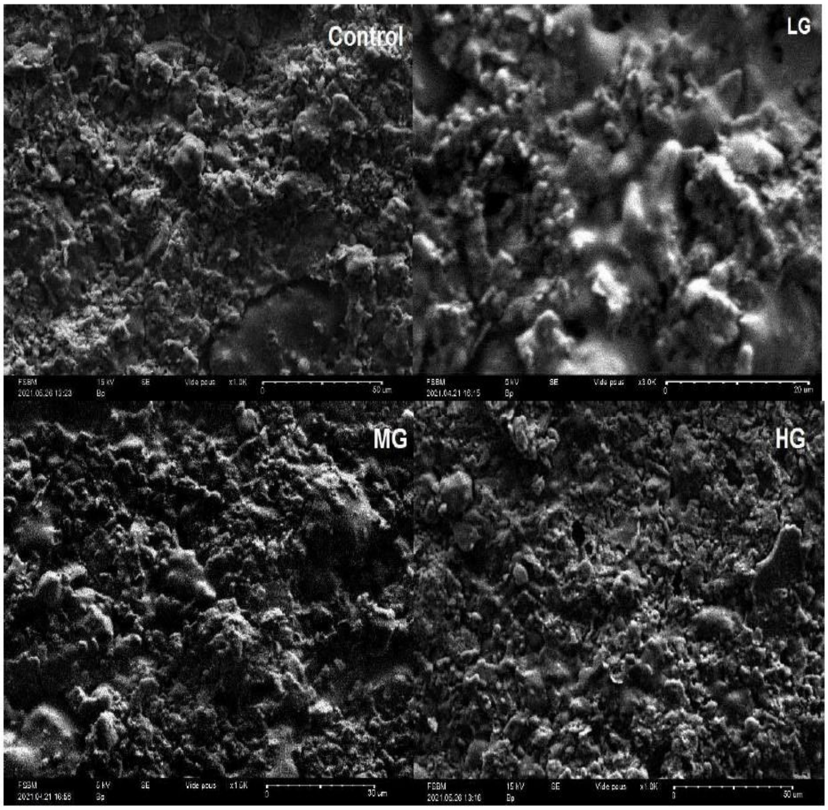

Figure 6. MEB observations of the adhesion of *E. coli* on the different surfaces of the tested materials

4. Discussion

Improving mechanical and technical quality of ceramic products using specific additives has also been opted for by several authors in the literature. Sawadogo *et al.* (2014) and Amin *et al.* (2019) used talc to improve the technical properties of tiles reducing investment in terms of energy consumption. Yang *et al.* (2017) and Yonghao *et al.* (2017) showed the effect of mining waste rock from phosphate mining activity in China, demonstrating the significant impact on water absorption and technical properties. A similar study by Moukannaa *et al.* (2018), valorizing phosphate sludge produced in large quantities during phosphate ore beneficiation mixed with metakaolin to produce geopolymers led to reinforced and dense materials with good mechanical properties. The mechanism of the impacts on the recorded shrinkage, absorption and mechanical properties has been mentioned by some researchers (Scrivener et al., 2015; Zheng et al., 2015). More specifically the study conducted by Scrivener *et al.* (2015), and Zheng *et al.* (2015), have shown the feasibility of using residues in Portland cement as a filler and explains the imparts on setting time, resistance, and drying shrinkage mainly by the dilution effect. Dissolution of phosphorus in the material has a slight retarding effect on hydration, dissolved phosphorus prolongs the induction period, reduces the main heat peak by the precipitation mechanism. In this study, the effect of integration of phosphate products of different grades on technical performance depends on the concentration and the nature of the grade of phosphate involved in the formulation. More generally, the phosphate content acts, probably, favorably on the mechanical properties of ceramic tiles with almost no effect on water absorption. The weight shrinkage seems to be reduced with the integration of components rich in P_2O_5. The integration of phosphate products in the formulation improves the mechanical properties of the tiles. Materials that engage either MG or HG at least 15% meet technical requirements. From an anti-adhesion and biofilm-forming standpoint, phosphate products have been studied by several authors for their interesting effects in improving hygiene and treating pollution. Nie *et al.* (2020) propose an efficient and environmentally friendly process for the reduction of SO_2 (desulfurization) using phosphate tailings as an adsorbent. Weng *et al.* (2003), Huang and Nguyen *et al.* (2015); Hakou *et al.* (2016) have shown the efficiency and capacity of residues and different forms of phosphate in my multimetal depollution, adsorption thus in improving hygiene. This study shows that the integration of phosphate products in the formulation improves the hygiene of ceramic tile surfaces and prevents the adhesion and formation of biofilms. Indeed, adhesion tests show a considerable reduction in bacterial growth, which is a function of the P_2O_5 content and may reach up to 75%. Previous research studies have shown that the adhesion of bacteria to surfaces depends on the nature of the material (Ferraz et al., 2004; Boutaleb et al., 2008b, El Omari et al., 2017, El Omari et al., 2018). When the material has no bioactive properties, its physico-chemical properties (e.g. hydrophobicity, acid-base character, electrostatic properties) are the determining factors of surface-bacteria affinity, and bacteria are capable of regulating their surface properties to resist against environmental stress. However, this property, can govern and mask the effect of physico-chemical interactions, also called non-specific when material has bioactive properties (Ferraz et al., 2004; Boutaleb et al., 2008b; El Yakoubi et al. 2006; El Omari et al. 2018).

Conclusion

This study proposes a solution to improve the mechanical and hygienic properties of ceramic tile products and prevent bio adhesion and the formation of biofilms by using phosphate products as an additive in the formulation. It has been shown that the effect on both mechanical and hygienic properties is proportional to the content of P_2O_5 in the additive. This study shows that the gradual addition of the phosphate-rich additive helps: (1) reduces weight loss with a more or less stable loss in diameter; (2) does not significantly influence the water absorption; (3) the addition of at least 15% of Medium Grade and 15% of Hight Grade of phosphate product from Morocco is necessary, to meet ISO requirements and also gives the ceramic material anti-biofilm properties; This proposed alternative is ecological and inexpensive, it makes possible to improve the durability and hygiene of ceramic surfaces by combating the formation of biofilms, without affecting the other required technical properties. However, it is important to validate all the results obtained in the real environment with which the tiling material would be confronted, and also to see how the anti-bioadhesion effect evolves over time.

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Conflict of Interest: All authors declare no conflict of interest.

Ethical approval: This study was conducted in full accordance with ethical principles. All experimental protocols were carried out by the relevant guidelines and regulations.

Competing interests: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability statement: The data that support the findings of this study are available from the corresponding author upon reasonable request.

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