



Content of crystalline silica phases in porcelain stoneware

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ABSTRACT

Kitchen and bathroom countertop is a demanding application, where high aesthetic standards must combine with durability, ease of maintenance, and resistance to heat, stain, scratch and chipping. The hard materials and composites used for high-end countertops usually contain crystalline silica phases that can be inhaled by workers during drilling and cutting operations. The occurrence of silicosis and other respiratory diseases in machining workers makes it important to know exactly how much crystalline silica is present in countertop materials. This paper collects over 300 quantitative determinations of quartz and cristobalite in porcelain stoneware products and compare these contents with other countertop materials. The sum of crystalline silica phases in porcelain stoneware is on average $21 \pm 5\%$ by weight (mostly quartz). This content is lower than granite ($\sim 30\%$) and much lower than engineered stone ($\sim 90\%$). Possible ways to reduce the amount of crystalline silica phases in ceramic slabs are overviewed and critically discussed.

1. Introduction

Various materials currently meet the demand for kitchen and bathroom countertops: ornamental stones, including granite, marble, soapstone, etc. [1]; quartz-resin composites, also called engineered or agglomerated stone [2,3]; ceramics, i.e. porcelain stoneware [4,5]; solid surface composites, based on aluminium trihydroxide [6,7]; concrete and wood [8]. These materials must fulfil several performance requirements, the most important being the resistance to heat, stain, scratch and chipping, along with ease of maintenance [9]. All these requirements, combined with remarkable durability and high aesthetic standards, are satisfied in the most demanding applications – which include, together with countertops, floating floorings, interior and outdoor coverings, and various furnishings – by hard materials, such as granite, engineered stone or porcelain stoneware [9,10].

The application of countertops requires, especially in the case of high-end materials, mechanical processing for drilling, cutting, grinding, etc. These operations are usually carried out in dedicated processing plants or sometimes directly on site, during the countertop installation [11,12]. Cutting and drilling operations produce dusts that, in the absence of adequate powder abatement and extraction systems, can be inhaled by workers, since a fraction of them have the particle size characteristics of respirable dust [12,13].

There is growing concern about workers' exposure to countertop processing dusts because they contain respirable crystalline silica

[14–17]. Recently, cases of silicosis [18–21] and other respiratory diseases [17] have been recorded in various countries in countertop machining workers. In some countries this situation is leading to legislative measures to limit workers' exposure to respirable crystalline silica, that are specific for countertops and similar applications [22,23]. To this purpose, directives on carcinogens and mutagens have been implemented in the ceramic production lines (for example, the European Directive 2017/2398 has been introduced into the scope of the so-called Directive CMD - 2004/37/EC).

In 2021 the Memorandum of Understanding between the Emilia Romagna Region - Acimac - Confindustria Ceramica and the trade union organizations have defined criteria for the ceramic industry for the identification of jobs involving exposure to respirable crystalline silt generated by a manufacturing process, referring to an “exposure limit value of 0.1 mg/m^3 measured or calculated in relation to a reference period of 8 h for the dust generated by the material processing”. For this reason, it is important to know exactly the crystalline silica content in countertops, especially for high-end products made up of hard materials. In many cases, however, the composition is defined at the time of product formulation (e.g., engineered stone) since the manufacturing process does not reach temperatures so high as to transform the inorganic phases present [2,3], in specific crystalline silica phases (quartz or cristobalite). In the case of ornamental stones, on the other hand, the composition is known for each igneous lithotype [24,25] or granite deposit, as prescribed by standards (e.g., EN 12407:2007 and ASTM

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C1721-21a) and often available in inventories and databases (e.g., Refs. [26,27]).

At variance, in the case of porcelain stoneware, the crystalline silica content is generally not known for several reasons: at the batch design level, it is not trivial to calculate the actual quartz content, since it is ubiquitous in almost all ceramic raw materials [28,29]. Most importantly, quartz is transformed to a certain extent during the firing process, because of complex reactions that depend on various factors [30,31]. Unfortunately, it is not straightforward to predict reliably how much quartz will be melted or whether cristobalite or tridymite will be formed [32]. Therefore, it is necessary to quantify accurately the content of quartz (and other silica phases) in the finished product, considering that such a determination requires a destructive technique, which is affected by sample preparation and needs specific procedures [33,34].

The aim of this work is the critical review of crystalline silica data in porcelain stoneware and comparison with other materials used for kitchen and bathroom countertops. In addition, possible ways to reduce the quartz content, their efficacy and technological readiness will also be assessed and discussed.

2. Data collection

Data on crystalline silica content in porcelain stoneware were collected through an exhaustive literature search, corroborated by unpublished results. A total of 206 samples of standard porcelain stoneware bodies were taken into consideration, of which 97 were commercial products and 109 were equivalent batches made in the laboratory (Fig. 1). In addition, 97 samples of innovative porcelain stoneware formulations were considered, in which conventional raw materials were partially replaced by strong mineral fluxes (e.g., nepheline syenite, diopside, or spodumene) or glass fluxes (e.g., soda-lime cullet) or waste materials (e.g., combustion ash). Bibliographic references of the entire set of 303 porcelain stoneware samples (including 20 unpublished data) are given in Table 1S (supplementary material).

Data refer to ceramic manufacturers (commercial products or laboratory replicas) in different countries: Italy (202 samples), Turkey (39), Brazil (24), Spain (19), China (13), India (3), Argentina (2) and South Korea (1).

Determination of crystalline silica phases (quartz, cristobalite, tridymite) was performed – according to the experimental description of the various papers – by X-ray powder diffraction. In most cases, a full profile Rietveld refinement of the XRD pattern was carried out by means of specific software packages (GSAS, TOPAS, MAUD, FullProf). In few cases, quartz and cristobalite were quantified by single peak approach (RIR method). These analytical techniques allow to determine crystalline silica phases with accuracy and precision within 1 % by weight.

Unpublished data regard both commercial and laboratory porcelain stoneware bodies, analysed by X-ray powder diffraction (Bruker, D8 Advance and LynxEye, Karlsruhe, Germany). Patterns were collected

Table 1

Content of crystalline silica phases (quartz, cristobalite, tridymite) in porcelain stoneware and other hard materials for countertops.

Material	Average (% weight)	Range (% weight)
Porcelain stoneware (standard)	~21 %	10–32 %
Engineered stone (quartz-resin composites)	~90 %	80–94 %
Granite (ornamental stones)	~30 %	16–42 %

with a Cu X-ray tube (operating at 40 kV and 40 mA) from 10 to 100°2 θ , step size of 0.02°2 θ , counting time of 1 s per step. Every sample was admixed with 20 wt% corundum as internal standard for the quantification of crystalline and amorphous phases [33,35]. The XRPD patterns were modelled by a Rietveld refinement with the TOPAS software [36].

3. Crystalline silica content

In porcelain stoneware slabs, crystalline silica is always present as quartz [5,37–104]. At variance, cristobalite was found in approximately 9 % of samples [44,55,66,79,88] and tridymite only in one sample [88].

The sum of crystalline silica phases in standard porcelain stoneware is on average 21.0 % \pm 5.2 %. This figure substantially confirms previous assessments [61,102]. In particular, quartz is on average 19.7 % \pm 5.6 % and cristobalite 2.0 % \pm 1.5 %. The amount of crystalline silica phases in commercial porcelain stoneware (21.7 % \pm 5.6 %) is substantially the same that in laboratory-made samples (20.3 % \pm 5.7 %) and the two populations are undistinguishable from the statistical point of view (Fig. 2A).

Ceramic technology is versatile enough to allow the use of a wide range of raw materials and this reflects in a certain degree of freedom in the design of porcelain stoneware batches [4,28]. Despite this, there are no statistically significant differences between manufacturers in various countries (Fig. 2B). The figures relating to Italian production are robust, as they come from a high number of industrial products, while those from other countries have a narrower sample base and are therefore statistically less reliable.

It is then possible to take the overall content of crystalline silica phases in porcelain stoneware and compare it to other materials for countertops, in particular those used for high-end applications (Table 1). Both the average value of quartz + cristobalite in porcelain stoneware (21 %) and its range (10–32 %, as 95 % of data distribution) are lower than the corresponding values usually found in granitic rocks, where only quartz occurs [24,25] and much lower than typical contents of engineered stones, where both quartz and cristobalite are commonly used [2,3,12–14].

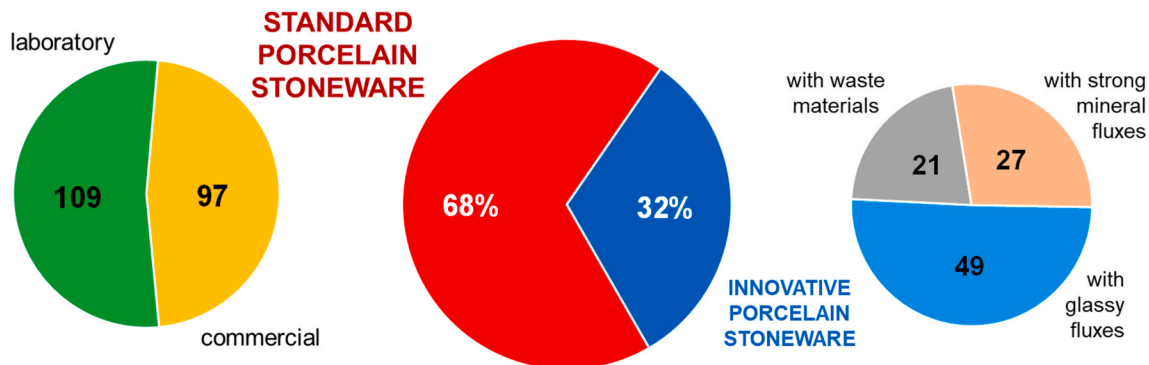


Fig. 1. Sampling of porcelain stoneware bodies.

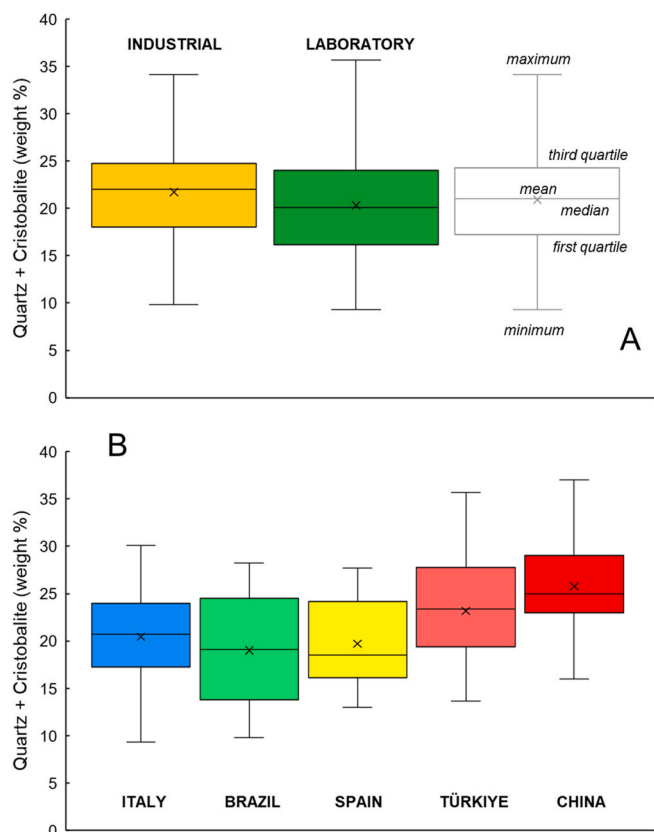


Fig. 2. Box-and-whisker diagram of the crystalline silica content (quartz + cristobalite) in standard porcelain stoneware: A) commercial (industrial) versus laboratory made bodies; B) bodies from various countries.

4. Reducing the content of crystalline silica phases in porcelain stoneware

The presence of crystalline silica phases in porcelain stoneware bodies is essential for some technological functions:

- To control indirectly (by the so-called tempering effect) the rheological behaviour of slips, the compaction of the green body and prevent deformations during drying [105,106];
- To control (by the so-called skeleton effect) the firing shrinkage and prevent excessive permanent deformations or breakage during sintering [107,108];
- To provide a spontaneous mechanism of high temperature bulk viscosity control (by the so-called buffering effect) being able to compensate for effective viscosity loss with increased melt viscosity in case of quartz melting [46,109];
- To increase the coefficient of thermal expansion of fired bodies, entailing at the same time the risk of rupture during cooling due to the β - α transition of quartz [110,111].

An overview of possible ways to reduce the amount of crystalline silica phases in porcelain stoneware is described below. A possible strategy to be undertaken has been defined in order to outline the feasibility and possible obstacles to be overcome.

4.1. Decreasing the content of quartz in the formulation of the ceramic body

Quartz is present in almost all ceramic raw materials (clays, feldspathic fluxes, silica sands) although its actual content is not always considered in batch design practices [28]. Selecting the plastic and/or

flux components with a low quartz content entails the choice of peculiar raw materials [112], which are presumably more expensive. In this way, any reduction of quartz in the raw batch does not automatically result in a corresponding decrease in the content of the crystalline silica phases after firing. Although there is a broad positive relationship between the silica content of raw materials and the amount of quartz in the finished product, this suffers from strong uncertainties (Fig. 3A). This path is certainly to be followed, limiting the quartz content at the beginning, but does not allow an accurate prediction of the final content of the crystalline silica phases.

4.2. Greater reactivity of quartz during firing

It is known that porcelain stoneware has a complex evolution – starting from feldspars, clay minerals and quartz of raw materials – which react with each other during the thermal cycle [30,34,46,71]. Quartz can be involved in the chemical transformations that take place at high temperature [31] depending on various factors, even if particle size and firing time are the major variables [32,61]. The fraction of quartz that is lost during firing varies widely, without a clear correlation with the silica content, so that for 71–72 % of silica in the body, the reduction goes from zero to 50 % (Fig. 3B). As a matter of fact, predicting how much quartz will be melted in vitrified ceramics and whether polymorphic phases (cristobalite or tridymite) will form is not a trivial task. An example is porcelain stoneware bodies with a different silica concentration, as shown in Fig. 4, where laboratory and industrially prepared samples are plotted in the SiO_2 - Al_2O_3 /(Al_2O_3 + $\text{Na}_2\text{O}_{\text{eq}}$) diagram [45]. Although the industrial bodies have on average a higher silica content than the laboratory ones, the amount of quartz remaining after firing is substantially the same, from the statistic point of view, even if the average value differs a little (Fig. 2A).

Nevertheless, it was often observed that the introduction in porcelain stoneware formulations of glassy raw materials (or some strong fluxes) can induce greater reactivity of quartz during firing [42,45,48,67,72,96,101,113–115]. Indeed, if ordinary bodies are compared with porcelain stoneware formulations containing glassy raw materials or strong fluxes, it can be appreciated a lower quartz content in the latter (Fig. 5). These unusual formulations give rise to peralkaline melts, where quartz appears to have faster dissolution kinetics [45,48]. However, it should be considered that the technological properties of bodies containing glassy raw materials are not the same as standard porcelain stoneware and that a deterioration in the degree of densification and pyroplasticity was observed [45,96].

The addition of residues as raw materials in porcelain stoneware batches has become a common industrial practice [82,88,116]. However, considering waste-bearing tiles, it cannot be noticed significant changes in the final content of crystalline silica phases compared to standard stoneware porcelain (Fig. 5).

The role of quartz grain size in its dissolution kinetics must also be taken into account, even though there are few specific studies on porcelain stoneware bodies [61,111,117]. The main limitation is that most available data concern the particle size distribution of the mixture (and not that of quartz). This seems to be an effective approach: quartz decreased approximately from 24 % to 20 % when particle size was reduced from 20 to 5 μm [61]. However, such diminution is still far from a target of crystalline silica phases below 10 %. A key issue is that particle size distribution is an important technological constraint for compaction and firing behaviour [105,118]. It is not by chance that industrial particle size curves tend to draw in a rather narrow range. The degrees of freedom for changing the grain size of quartz are therefore limited and concern more the use of micronized raw materials than any change in the milling schedule.

Overall, improving the quartz reactivity during firing is a promising way to reduce the amount of crystalline silica phases in porcelain stoneware tiles. Nonetheless, it is a solution that requires careful design, as it involves major changes in batch formulation and affects relevant

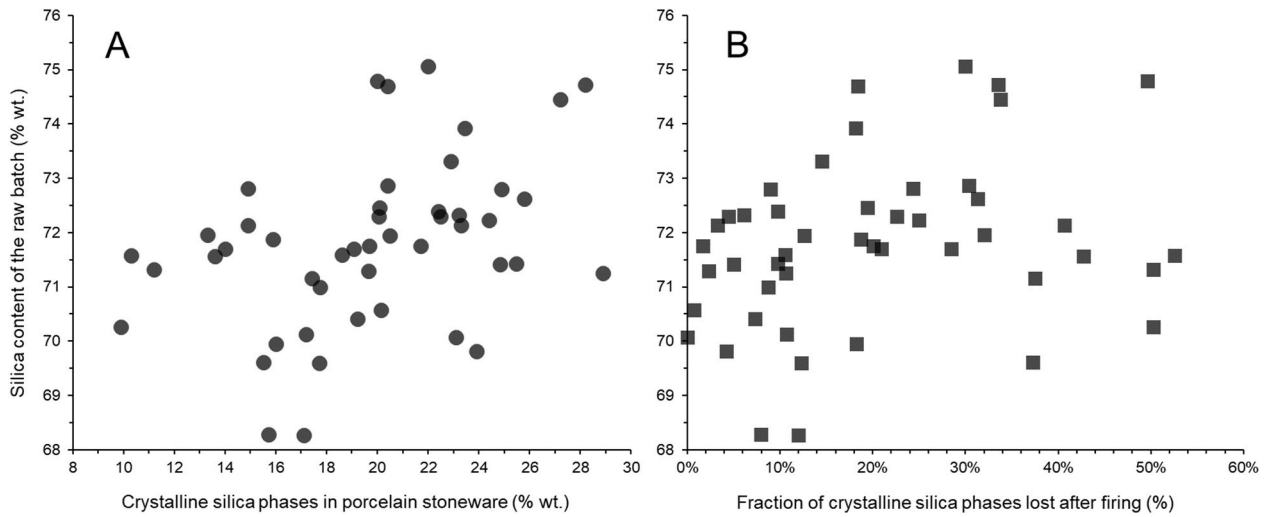


Fig. 3. Content of crystalline silica phases (quartz + cristobalite) in standard porcelain stoneware (A) and fraction of crystalline silica phases reacted and lost during firing (B) versus the SiO₂ amount of the raw batch.

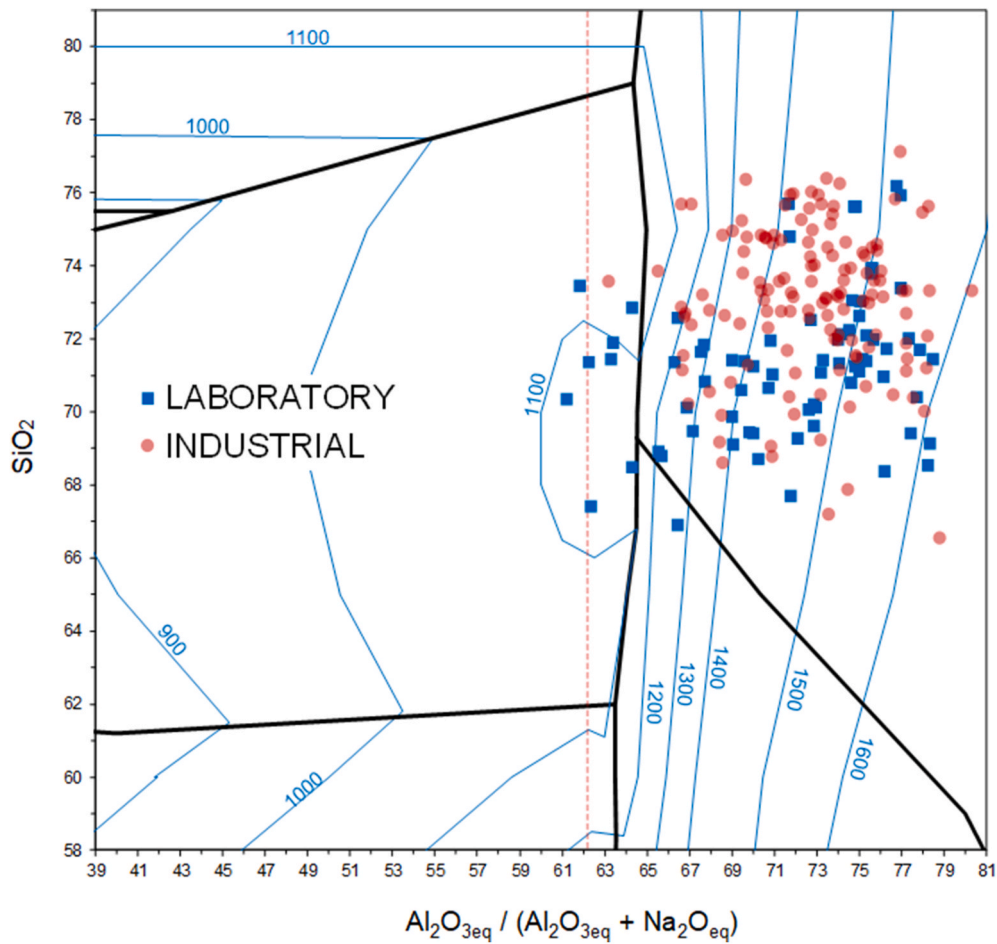


Fig. 4. Chemical composition represented in the binary graph silica-alumosity [Al₂O₃/(Al₂O₃+Na₂O_{eq})].

technological issues mentioned above, such as the skeleton and buffering effects.

4.3. Search for alternative fillers

It is necessary to look for suitable materials that can even partially

replace the crystalline silica phases in porcelain stoneware bodies, which can act as filler (ensuring the tempering and skeleton effects). This implies that candidates must have dissolution kinetics during firing comparable to quartz. In particular, the alternative crystalline phase should also ensure some buffering effect, i.e., keeping the effective viscosity of the ceramic body in the event of dissolution of the filler. In

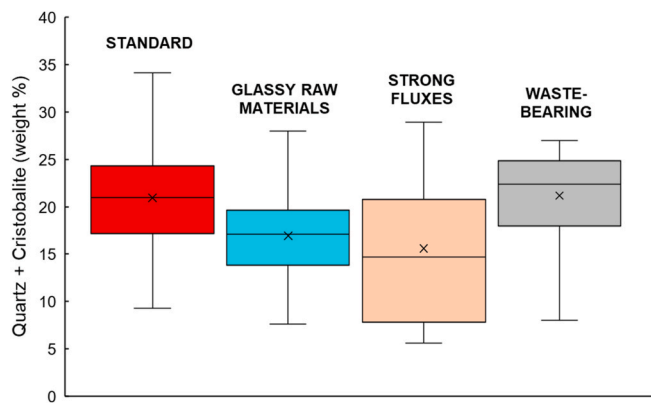


Fig. 5. Box-and-whisker diagram of the crystalline silica content (quartz + cristobalite) in standard porcelain stoneware in comparison with innovative formulations containing unusual raw materials.

addition, suitable fillers must have the coefficient of thermal expansion high enough to meet the standard requirement for porcelain stoneware slabs (that anyway was based on the thermal properties of quartz).

However, it should not be overlooked that the replacement of quartz would lead to the disappearance of the β - α phase transition during cooling, which involves a significant volume variation and the remarkable risk of damaging the porcelain stoneware countertops [111,117].

A partial replacement of quartz with zircon has been industrially demonstrated in super white porcelain stoneware bodies [44,91,119] but it has to face limitations stemming from cost and radioactivity of zirconium silicate [120,121]. In the literature, other opacifiers have been successfully utilized on a laboratory scale, such as alumina [40, 122] or glass-ceramics frits [101,123]. White-firing bodies, based on anorthite and/or diopside, have also been developed for low silica porcelain stoneware tiles [92,124–126].

This way seems to be the most effective in reducing the content of crystalline silica phases in porcelain stoneware countertops but must overcome the limitation of increasing costs. From a technological point of view, there is no overall picture of pros and cons for different candidates (especially for glass-ceramics) to replace quartz as filler in the ceramic body.

4.4. Combining batch design and quartz reactivity during firing

The combination of the options described above to reduce the content of crystalline silica phases in porcelain stoneware tiles should have greater effectiveness than individual actions. Although there are apparently no cross-constraints to lower the initial percentage of quartz and increase its reactivity during firing, the overall technological behaviour of the ceramic body must be kept within the standard requirements. This is a complex task, as it entails, on the one hand, to select suitable quartz substitutes and/or raw materials with a low quartz content and decrease the particle size (e.g., by using micronized fluxes). On the other hand, it needs that reactive melts form (by adding glassy raw materials or strong fluxes into the batch) where alternative fillers should be stable.

This combination seems to be the most promising way to have a consistent reduction in the quartz content in porcelain stoneware slabs (and so reach the target of crystalline silica phases below 10 % weight). However, there is not enough knowledge to predict whether feedback from one action on the other (e.g., the buffering effect at high temperature) can be minimized or represent a challenging obstacle.

More research and development is needed on the combination of the various technological solutions to reduce the content of crystalline silica phases in the production of countertops in porcelain stoneware.

4.5. Glass-ceramic composites

A well-known technology for obtaining vitrified silicate materials is the glass-ceramic route, which consists of batch melting, followed by controlled crystallization during cooling [127,128]. Although glass-ceramics exhibit a high degree of densification and excellent mechanical-tribological properties, descending from accurate micro-structural design [129,130], they suffer from limitations about the product dimension (much smaller than the market demand for countertops) and manufacturing cost (one order of magnitude higher than ceramic tiles).

An alternative route – somewhere halfway between glass-ceramics and ceramic countertops – is the sintering of batches consisting of glass (as a raw material) and filler (quartz or other crystalline compounds capable of withstanding at high temperature the chemical attack of the liquid phase). This technology is less widely known than glass-ceramics [131,132] particularly as a manufacturing route for countertop materials. Recently, explorative work was published on composites consisting of waste glass (soda-lime cullet) sintered together with quartz, albite, petalite or corundum as filler [133]. The results obtained are encouraging – except in the case of quartz – even though the technology has so far been developed only for small sizes and at a low readiness level [132,133].

5. Conclusions

The sum of crystalline silica phases in porcelain stoneware is on average 21.0 % \pm 5.2 % by weight, with 95 % of products within the 10–32 % range. Quartz is predominant over cristobalite (on average 2.0 %) that was found in approximately 9 % of samples. In comparison with other materials for countertops, and in particular those used for high-end applications, the content of crystalline silica phases in porcelain stoneware is lower than the values usually found in granitic rocks (~30 %) and much lower than typical contents of engineered stones (~90 %). Four ways to reduce the amount of crystalline silica in porcelain stoneware have been discussed to outline feasibility and possible obstacles to overcome:

Possibility of decreasing the quartz content in the formulation of the ceramic body: given that quartz is present in almost all ceramic raw materials, its reduction in the raw batch does not automatically lead to a corresponding decrease in the content of the siliceous phases crystal after firing. This is certainly a viable path but an accurate prediction of the final content of the crystalline siliceous phases is not possible.

Promoting greater reactivity of the quartz during firing: improving the reactivity of quartz during firing is a promising solution to reduce the amount of crystalline silica phases in porcelain stoneware tiles. However, important changes must be implemented in batch formulation and technological aspects (for example skeleton effects and buffering) must be taken into consideration.

Looking for alternative fillers: candidates must meet several technological requirements (dissolution kinetics, effective viscosity, thermal expansion coefficient) along with cost. White-firing bodies, based on anorthite and/or diopside, for porcelain stoneware tiles with low silica content can be an effective solution for reducing the content of crystalline silica phases in porcelain stoneware countertops, but they are expensive.

Combining batch design and the reactivity of quartz during firing seems to be the most promising option for obtaining a consistent reduction in the quartz content in porcelain stoneware slabs. However, further research and development is needed as there is not enough knowledge to predict whether feedback from one action to another can be minimized or represent a challenging obstacle.

Data availability

The raw/processed data required to reproduce these findings cannot

be shared at this time as the data also forms part of an ongoing study.

CRedit authorship contribution statement

Chiara Molinari: Writing – review & editing, Writing – original draft, Visualization, Validation, Data curation, Conceptualization. **Sonia Conte:** Writing – review & editing, Writing – original draft, Methodology, Data curation, Conceptualization. **Michele Dondi:** Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Chiara Zanelli:** Writing – original draft, Validation, Supervision, Resources, Project administration, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.oceram.2024.100650>.

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