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Original Research / Orijinal Araştırma

Use of marble dust containing filler mixture in composite slab production: Relationship between roasting conditions and physical properties of the slab

Mermer tozu içeren dolgu karışımının kompozit plaka üretiminde kullanımı: Kavurma koşulları ile plaka fiziksel özellikleri arasındaki ilişki

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A B S T R A C T

Marble dust generated during slab cutting as reject causes significant environmental problems due to increased reactive surface area. It has closer size distribution with micronized quartz, which is used as filler in composite slab production. Owing to its high hardness, micronized quartz production is an energy intensive process. This study was conducted to investigate the applicability of marble dust in composite slab production together with micronized quartz as filler. The filler mixture was roasted to mitigate drawback arising from low hardness of marble dust. XRD results revealed that phases in roasted filler were wollastonite, larnite, calcio-olivine, quicklime and free quartz depending on the roasting temperature and time. Physical tests were applied to clarify the effect of sinter phases on slab properties. Physical properties were determined to retrogress as the roasting temperature increased to 1100°C possibly due to rate of free lime in roasted filler, and then improved again reaching peak point at 1200°C. Physical properties ameliorated by increasing roasting time at 1200°C. Larnite and quartz were found to be effective on improved physical properties than wollastonite and calcio-olivine.

Keywords: *Composite slab, Micronized quartz, Marble dust, Roasting*

Introduction

Advances in civilization, and in turn, development in building industry have led to considerable increase in the consumption of natural stones, like marble and granite. Marble is generally consumed as countertops, floor tiles, wall tiles, steps and table tops in the form of slab (Albalak, 2012). Marble cutting process into slab form generates finely sized marble dust (MD) (Gazi et al., 2012; Güler and Polat 2018). The rate of MD generated during slab cutting varies volumetrically from 15% to 40% of marble block depending on the morphology, mineralogical composition and crystalline structure of the rock, type of slab cutting machine (gang saw or disc sawing) and slab thickness (Erdem and Öztürk, 2012; Liguori et al., 2008).

Slab cutting is performed in wet condition. Therefore, the generated MD is taken in the form of sludge containing approximately 35-45% water. The marble sludge is subjected to solid-liquid separation by thickening, and then by filtration. Dewatered MD is stored in waste disposal areas in the form of filter cake (Kocabağ, 2018). MD exposes significant environmental risks due to reasonably reactive high surface area. It may give rise to aridity around disposed land, reduce soil fertility, adversely affect the breathable air quality due to pollination, and affect negatively the eco-system (Kocabağ, 2018; Rana et al., 2016).

Research on high strength composite slab production has taken great interest owing to its high flexural strength and impact resistance in addition to low water absorption rate compared to marble slabs (Borsellino et al., 2009; Lam dos Santos et al., 2011;

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Santos et al., 2019; Tunç, 2021). Use of quartz both in aggregate (~66%) and micronized (~27%) forms induce the physical properties of composite slabs. Quartz particles are locked to each other by the aid of organic binders under vibration and vacuum pressure (Arıcı et al., 2019; Lam dos Santos et al., 2011; Peng and Qin, 2018). Quartz is a hard silicate mineral with the hardness value of 7 in Mohs scale. Therefore, preparation of micronized quartz as a filler component is an energy intensive process (de Bakker, 2014). Health threats (e.g., silicosis) of micronized quartz, and occupational concerns during production are other issues to be regarded.

The underlying idea in defining the aim of this research work was the possibility of MD usage as filler instead of micronized quartz in composite slab production from economic and environmental point of concern. MD is composed mainly of calcite, which is soft mineral with the hardness value of 3 in Mohs scale (Güler and Polat 2018; Velázquez et al., 2008). Borsellino et al. (2009) reported that MD adversely affected the mechanical behavior and porosity of the produced slabs referring to the physical characteristics of matrix forming calcite mineral. This problem was thought to be overcome by roasting the mixture of soft MD and hard micronized quartz. Sintering roasting of the mixture produces harder complex Ca-silicates ($xCaO.ySiO_2$) as compared to MD (Boonchareon et al., 2011; Klosek-Wawrzyn et al., 2013; Liu et al., 2002; Nettleship et al., 1993). Main complex phases are mono-calcium silicate - $CaO.SiO_2$ (wollastonite) and di-calcium silicates - $2CaO.SiO_2$ (larnite and calico olivine) depending on the rate of roasting time, roasting temperature and CaO/SiO_2 molar ratio. Wollastonite ($CaSiO_3$) forms at lower molar ratios while metastable larnite (β-Ca_2SiO_4) and stable calcio-olivine (γ-Ca_2SiO_4) phases were the dominating reaction products at higher rates after extended roasting process (Akaogi et al., 2004; Gobechiya et al., 2008; Göktaş and Erdemoğlu, 2012; Lakshmi et al., 2013; Rashid et al., 2014; Zadov et al., 2008). Larnite is the heaviest dicalcium silicate phase among them with the density of about 3.30 g/cm^3 while this value is 2.99 g/cm^3 for calcio-olivine, and around 2.90 g/cm^3 for wollastonite. Hardness of wollastonite is between 4.5-5.0 in Mohs scale while it is 6 and 4.5 for larnite and calcio-olivine, respectively (Joesten, 1977; Liu et al., 2002; Nettleship et al., 1993). Variation in the shape of sintered particles is another advantageous property of thermal process. Roasted particles tend to shape into globe as a result of cohesive forces by partial softening of particle surfaces. So, roasting releases rounded sinter particles, which improves the mechanical properties of produced material (Miyake et al., 2018; Tunç, 2021).

Sintering roasting of filler mixture (MD + micronized quartz) was investigated to clarify the impact of sinter phases on the physical properties of composite slab. Variation in the rate of sinter phases in the roasted filler with respect to the temperature and duration of roasting process was demonstrated. Role of MD percentage of the filler on phase distribution in the roasted filler, and in turn mechanical properties and compactness of produced slabs were also studied.

1. Materials and methods

MD was supplied from Yatağan Marble Processing Plant of Ermaş Mining Company in the form of marble sludge while micronized quartz filler was taken from Coante Quartz Surfaces Plant of the same company. Characterization of filler materials was performed by X-ray diffraction (XRD), X-ray fluorescence (XRF) and size analysis. Chemical analysis held by XRF (PANalytical Axios Max) showed that CaO grade of MD was 55.92% assigning to a calcite content higher than 99% while micronized quartz filler contains 98.05% SiO_2. The XRD analysis was performed using SmartLab Rikagu XRD equipment by Cu-K_α radiation with a step size of 0.02° to determine mineralogical composition of fillers. Mineralogical characterization showed that fillers were highly pure: detected crystalline phase in MD was calcite, and that was quartz mineral in micronized quartz filler (Figure 1). Main identifying XRD peaks of quartz were at 2θ of 20.7° and 26.5°, and that was 2θ 29.5° for calcite (Boonchareon et al., 2011; Rohmawati et al., 2019). Size analysis of fillers was performed by Malvern Mastersizer. Fillers were measured to have closer size distributions: 90% of MD was at -46.5 µm while that was -42.2 µm for micronized quartz.

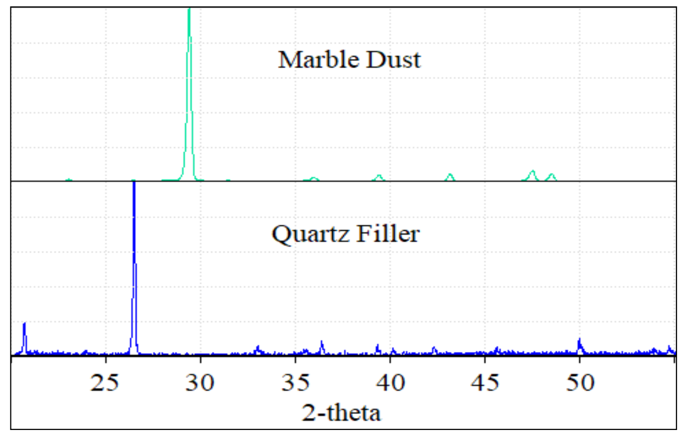

Figure 1. *XRD patterns of marble dust and micronized quartz filler samples*

MD substitution in slab production was investigated at different MD percentages as filler component, rest of which was micronized quartz. Filler mixture was prepared to satisfy even distribution of phases using a stainless-steel rod mill. Mixing was applied for 4 minutes with reduced rod charge (5%) to minimize comminution. Prepared filler mixtures were roasted in a muffle furnace (Ankatest). Roasting was applied at 900℃, 1000℃ and 1100℃ for 3 h, and at 1200°C for 1 h, 3 h, 5 h and 10 h to investigate the role of sinter phases on the physical properties of prepared slabs. Effect of roasting temperature was investigated only at 75% MD addition while roasting duration tests were performed on 25%, 50% and 75% MD containing filler mixtures.

As sintering roasting proceeds, adjacent sinter particles coalesce due to partial softening of particle surfaces, and consequently the total surface area decreases (Abenojar et al., 2003; Cardenas et al., 2016; Klosek-Wawrzyn et al., 2013; Rodriguez-Navarro et al., 2009). Therefore, rod milling was also applied on roasted filler with reduced rod charge (15%) for 4 minutes to break down weak physical bonds between particles, and to provide all particles pass through 45 µm sieve avoiding overgrinding. Rate of rod charge and milling time were determined by preliminary tests.

Composite slabs were produced using roasted mixture (-45 µm) as filler, coarse quartz (100-300 µm) as aggregate, and organic binders. Physical characteristics of produced slabs were investigated by applying flexural strength, impact resistance and water absorption tests. Details of the test procedure have been shared elsewhere (Arıcı et al., 2019; Callister and Rethwisch, 2018; Sarıışık et al., 2016; Tunç, 2021). Test results were compared with those of reference slabs (Table 1). Quartz composite slab (Product Code: STYL100320-1) of Coante Quartz Surfaces and marble slab (Muğla White) from Yatağan Marble Processing Plant of Ermaş Mining Co. were evaluated as reference slabs.

2. Results and discussions

2.1. Phase characterization of the filler roasted at 1200°C

Filler mixtures were roasted at 1200°C for different durations prior to usage in slab production. Phase identification of roasted fillers was made by XRD method. Figure 2 shows the X-ray reflections of roasted fillers in the 2θ range of 20-55° where pronounced peaks of new phases were observed. Roasting of MD containing filler mixture releases lime (CaO), which is also defined as quicklime. The CaO phase reacts further with quartz component of filler to form complex Ca-silicates. Resolution of XRD patterns showed that the lime phase drew major intense peaks at 2θ of 32.3°, 37.4° and 54.3° in the presence of high rate of MD while those for quartz were around 2θ 21° and 26.7° at lower MD percentages (Kostova et al., 2021; Mäkelä et al., 2011; Rohmawati et al., 2019; Witoon, 2011). XRD peaks on the pattern of 25% MD containing roasted filler mixture for 1 h overlaps almost with those on the pattern of micronized quartz given in Figure 1. The magnified view of peak structure of XRD pattern for 25% MD in the range 2θ 20°-40° was given on the figure since main identifying peaks of sinter phases could hardly be discriminated in the present form due to intense quartz peaks. New peaks appeared on this pattern especially between 2θ of 30°-35° when roasting time was increased to 3 h. Of which, the most intense one at 30.1° was assigned to wollastonite (Kostova et al., 2021; Tuttle and Harker, 1957). Wollastonite reflection deceased slightly at higher roasting times while identifying XRD peak of larnite phase at 2θ 22° became more apparent by extending the roasting process up to 10 h (Booncharoen et al., 2011; Kostova et al., 2021; Rashid et al., 2014; Tunç, 2021). Akaogi et al. (2004) proposed the phase transition as wollastonite ⇝ walstromite ⇝ larnite during complex calcium silicate formation from the mixture of $CaO+SiO_2$. Stable dicalcium silicate (calcio-olivine) phase formed by increasing MD percentage. The most intense calcio-olivine peak was obtained at 2θ of 29.6° (Booncharoen et al., 2011). Reflections drawn at about 23° and 28° were also explained with the formation of calcio-olivine. XRD peaks of lime and di-calcium silicates overlap around 2θ of 33° (Kostova et al., 2021; Mäkelä et al., 2011; Witoon, 2011).

Table 1. *Mechanical properties and water absorption rate of reference slabs*

	Flexural Strength MPa	Impact Resistance J/cm	Water Absorption %
Quartz Composite Slab (Coante Quartz Surfaces, Product Code: STYL100320-1)	77.21	3.96	0.0035
Marble Slab (Ermaş Mining Co., Muğla White)	10.14	1.42	0.1287

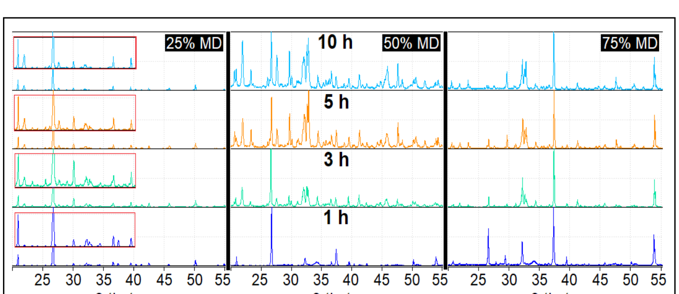

Figure 2. *XRD patterns of roasted filler mixtures (roasting temperature: 1200°C)*

Densities of roasted fillers were measured to determine the change in filler density with respect to the roasting time and the rate of MD (Figure 3). Minerals contained in the raw filler have closer densities, which one is 2.65 g/cm³ for quartz, and 2.71 g/cm³ for calcite (Moropoulou et al., 2001; Rashid et al., 2014). However, densities of detected new sinter phases vary in a slightly wide range: those are 3.34 g/cm³, ~3.30 g/cm³, 2.99 g/cm³ and ~2.90 g/cm³ for lime, larnite, calcio-olivine and wollastonite, respectively (Joesten, 1977; Liu et al., 2002; Nettleship et al., 1993). Roasting of 25% MD containing fillers gave rise to negligible increase in the density for all the roasting times tested as compared with the raw form of filler. Dominating phase detected was quartz while wollastonite and larnite were the new sinter phases for 25% MD. The roasted filler density was observed to be closer to quartz, which delimitated the effect of calcium silicates owing to volumetric distribution of phases. However, roasting time became more effective at higher MD percentages. Comparative evaluation of XRD patterns revealed that the rate of high density larnite and calcio-olivine increased at extended roasting whereas that of quartz decreased. High density larnite forms as a metastable phase during roasting. Extending the roasting causes the transformation of larnite to stable calcio-olivine phase having slightly lower density. Therefore, decrease in the roasted filler after 10 h roasting became apparent as seen in Figure 3 (Booncharoen et al., 2011; Gobechiya et al., 2008; Rashid et al., 2014; Zadov et al., 2008). Filler density shifted up considerably at 75% MD owing to the generation of high density free lime together with Ca-silicates when roasting was applied for 3 h or more. The filler density was low in case of incomplete roasting (1 h) of 75% MD containing mixture due to the presence of raw calcite and quartz phases as seen from XRD patterns.

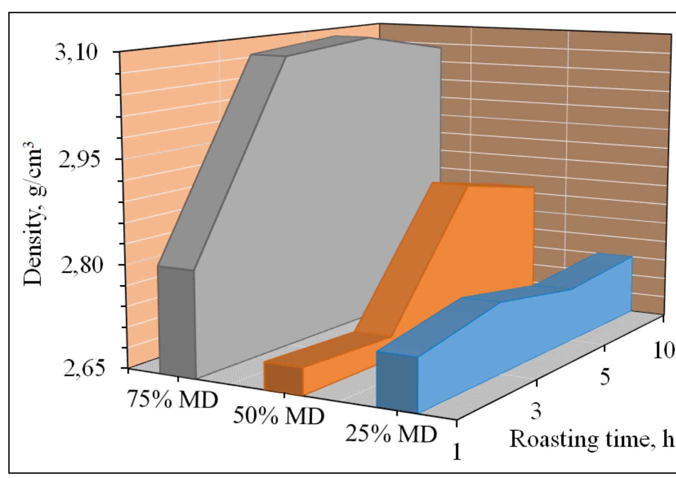

Figure 3. *Variation in the density of roasted filler mixture with respect to roasting time (roasting temperature: 1200°C)*

2.2. *Effect of roasting the filler at 1200°C on the physical properties of slabs*

Figures 4-5 show the effect of roasting time on the mechanical properties of the composite slab. As roasting time increased to 10 h, flexural strength (78.35 MPa) drove up for 25% MD, even over the reference point which was 77.21 MPa (Table 1) whereas slight decrease was observed for 50% MD (Figure 4). Similar trend curves were obtained for impact resistance of the slabs (Figure 5). It is worth noting that the impact resistance for 25% MD is conspicuously higher than that of reference quartz composite slab, but only for 10 h of roasting (4.34 J/cm). Mechanical data did almost not change for 75% MD except roasting for 1 h: these values were about 73 MPa and 3.50 J/cm for flexural strength and impact resistance, respectively. Average values of mechanical data for all the three tested MD rates were measured to be closer to each other with a slight increase at higher MD percentages. Prepared slab with MD containing roasted filler demonstrated remarkably improved mechanical properties reaching from 10.14 MPa and 1.42 J/cm to about 70 MPa and 3.2 J/cm for flexural strength and impact resistance, respectively.

Water absorption rate drew an inverse relation with respect to the mechanical properties of composite slab: it decreased by increasing the roasting time, where mechanical properties also improved simultaneously. However, proportional improvement in compactness could not be observed (Figure 6) Water absorption rate was appreciably low for 25% MD possibly due to the presence of quartz as the dominating phase. On the other hand, these curves were almost coinciding at higher MD rates although slight amelioration in the mechanical properties was observed by increasing MD rate. This finding was explained with the variation in phase distribution: the leading filler forming sinter phases were calcio-olivine and larnite together with lime for 50-75% MD containing roasted filler.

Mono calcium silicate (wollastonite - $CaO.SiO_2$) started to form in the initial stages of roasting of 25% MD containing filler mixture at 1200°C due to lower CaO/SiO_2 molecular ratio. Extended roasting of this filler caused the formation of metastable dicalcium silicate (larnite, β-Ca_2SiO_4) (Akaogi et al., 2004; Kostova et al., 2021; Lakshmi et al., 2013). XRD peaks of stable calcio-olivine (γ-Ca_2SiO_4) phase was not observed at lower CaO/SiO_2 rates (Figure 2) (Booncharoen et al., 2011; Tunç, 2021). Therefore, compactness of the slab for 25% MD was explained with the presence of quartz as the major filler phase in addition to the formation of wollastonite and larnite. Composite slab prepared by roasted filler for 10 h demonstrated appreciably better physical properties, even improved than reference quartz composite slab in which condition larnite was the sole defined Ca-silicate phase. Larnite is harder (around 6 in Mohs scale) than MD, but its hardness value was lower than quartz. So, improved mechanical properties of the slab was explained with the synergic effect of the rounded shape of particles, and hardness of sinter phase (Abenojar et al., 2003; Booncharoen et al., 2011; Cardenas et al., 2016; Klosek-Wawrzyn et al., 2013; Miyake et al., 2018; Rodriguez-Navarro et al., 2009). Sintering roasting cause partial softening of the surfaces of filler particles. These particles tend to coalesce during roasting due to cohesive forces.

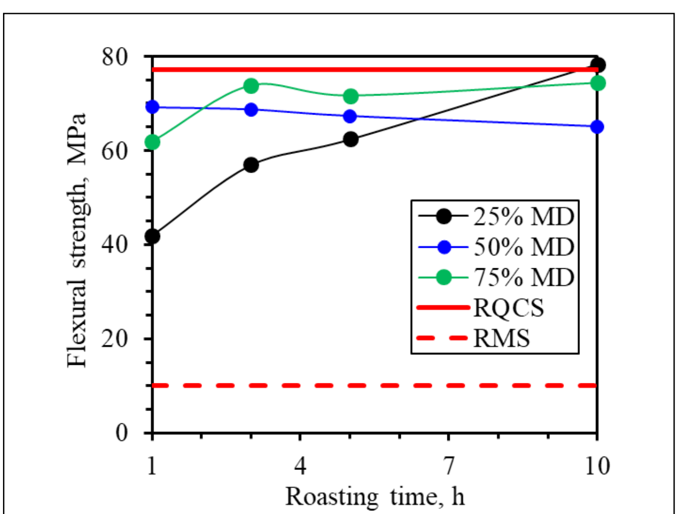

Figure 4. *Effect of roasting time on the flexural strength of composite slab prepared by roasted filler mixture (roasting temperature: 1200°C; RQCS: reference quartz composite slab; RMS: reference marble slab)*

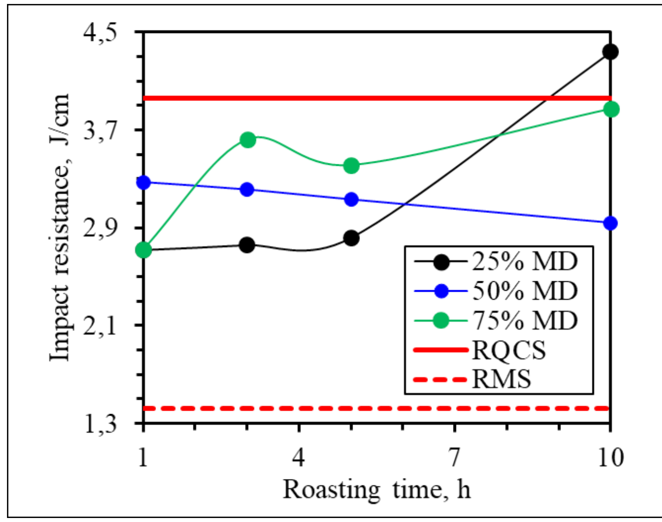

Figure 5. *Effect of roasting time on the impact resistance of composite slab prepared by roasted filler mixture (roasting temperature: 1200°C; RQCS: reference quartz composite slab; RMS: reference marble slab)*

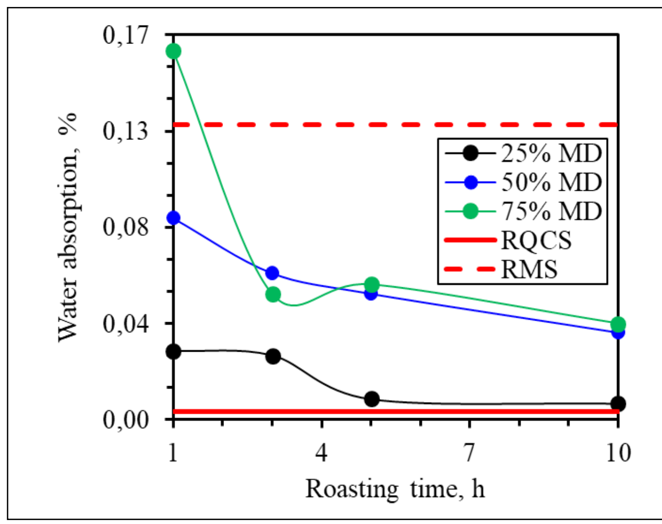

Figure 6. *Effect of roasting time on the water absorption rate of composite slab prepared by roasted filler mixture (roasting temperature: 1200°C; RQCS: reference quartz composite slab; RMS: reference marble slab)*

As a whole, closer mechanical values were obtained for all the tested MD percentages. The combined effect of sinter phases were thought to manipulate physical properties of slabs at different rates. The resolution of XRD patterns revealed that the rate of hard quartz phase decreased sharply for extended roasting periods in the presence of 50-75% MD in the filler mixture whereas percentage of stable calcio-olivine phase increased moderately (Tunç, 2021). Gradual decrease in the ratio of larnite phase was also observed from peak intensity evaluation. Sintering roasting produced quicklime together with the formation of complex dicalcium silicates at higher CaO/SiO_2 molecular ratios (Moropoulou et al., 2001). The quicklime (CaO) phase could not completely be converted into complex calcium silicates due to scarcity of SiO_2 in the roasted filler. Therefore, improved mechanical and water absorption properties of composite slab were attributed to the combined effect of CaO and considerably hard complex sinter Ca-silicate phases. However, deformation propagation in the slab produced by 75% MD containing roasted filler was observed 5 days later than slab production (Figure 7). Quantitative evaluation of the related XRD patterns revealed that the sinter products contain about 54% CaO and 28% CaO for roasting the filler mixture for 1 h and 10 h, respectively (Tunç, 2021). The deformation was thought to occur owing to the presence of CaO, and its conversion to slaked lime ($Ca(OH)_2$) under atmospheric condition according to following reaction (Kılıç and Anıl, 2005; Manzano et al., 2012; Moropoulou et al., 2001). Slaking was proposed to occur since CaO particles could not completely be isolated from atmosphere by polymers during slab production. Used polymer phase could not wet all the free reactive surfaces of particles due to its inadequacy in amount, which caused defectivity in the slabs.

$$CaO + H_2O \leftrightharpoons Ca(OH)_2 \quad (1)$$

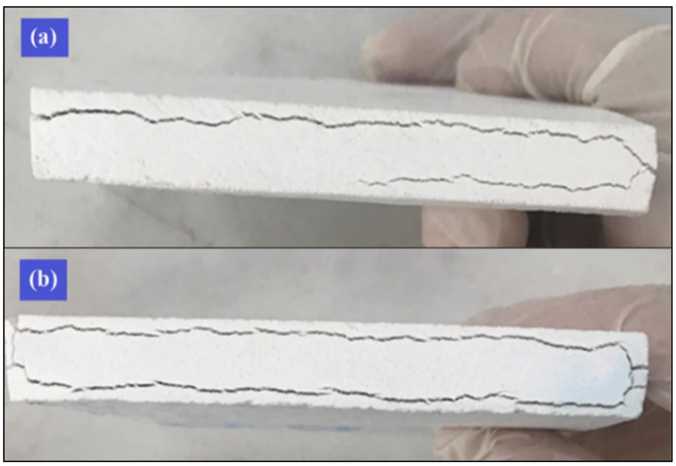

Figure 7. *Deformation of composite slab, 5 days after production for a) 1 h roasting and b) 10 h roasting (marble dust: 75%; roasting temperature: 1200°C)*

2.3. *Effect of roasting temperature on phase distribution and the physical properties of slabs*

Physical properties of the slabs, prepared by 75% MD containing roasted fillers, approach to those of reference quartz composite slab in spite of deformation. Therefore, the effect of roasting temperature on the phase distribution was investigated by the mixtures containing 75% MD. Figure 8 shows the XRD patterns of fillers roasted at different temperatures and durations. Main calcite reflection (29.4°) was observed only on the XRD pattern for 900°C indicating its presence in the roasted filler at a rate of about 36% according to quantitative resolution of XRD pattern by using Match software. In spite of incomplete calcination, appearance of sharp lime peaks at 2θ 37.4° and 54.3° was assumed as the reason for remarkably high density (2.87 g/cm^3) of the roasted filler (Kılıç and Anıl, 2005; Moropoulou et al., 2001; Rodriguez-Navarro et al., 2009). Above this temperature, calcite peak disappeared, and the carbonate phase was converted into oxide form.

The most pronounced peaks on the patterns show the presence of CaO as one of the major phases constituting the sinter product for all the tested roasting temperatures (Kostova et al., 2021; Mäkelä et al., 2011; Rohmawati et al., 2019; Witoon, 2011). The CaO percentage increases from about 28% to 60% by increasing the temperature to 1000°C, above which the rate of lime starts to decrease down to about 32% at 1200°C. Meanwhile, intensity of quartz peak at 2θ 26.7° deceased by increasing the temperature from 900°C to 1200°C showing the rate of change of quartz in the roasted filler from about 36% down to 9%. Increasing the roasting temperature resulted in a proportional increase in the intensities of dicalcium silicate peaks, especially those of larnite. But, all the CaO species could not be converted into dicalcium silicates due to scarcity of SiO_2 in the mixture (Booncharoen et al., 2011; Tunç, 2021). Proportional reflection of phase distribution on the densities of roasted filler was observed. It increased to 3.04 g/cm^3 at 1000°C by the contribution of CaO. Maximum density (3.12 g/cm^3) was reached at 1100°C due to the presence of lime at considerably high rate and absence of calcio-olivine. Generation of dicalcium silicates resulted in a slight decrease in density down to 3.08 g/cm^3 at 1200°C. The XRD reflections especially around 2θ 33° became sharpen at 1200°C by increasing the roasting time. The conversion of metastable dicalcium silicates (larnite) to stable one (calcio-olivine) caused this increase in the related XRD reflections (Akaogi et al., 2004; Booncharoen et al., 2011; Kostova et al., 2021; Lakshmi et al., 2013; Tunç, 2021). Calcio-olivine formation was also discriminated from the change in filler density. As seen from Figure 3, it decreased slightly from to 3.08 g/cm^3 to 3.06 g/cm^3 by phase change in consequence of the increase in the roasting time to 10 h at 1200°C.

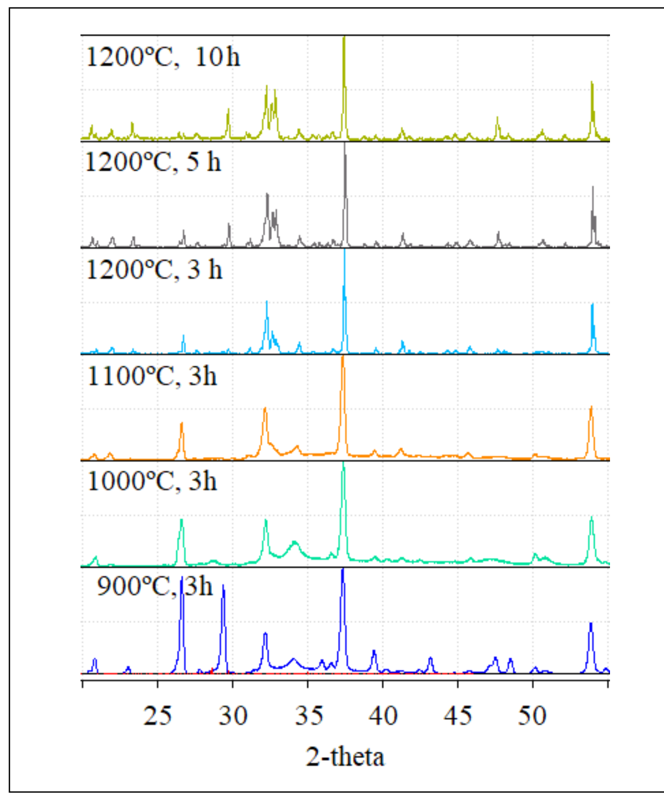

Figure 8. *XRD patterns of roasted filler mixtures (75% MD) at different temperatures and roasting times*

Figure 9 shows the variation in the physical properties of composite slab prepared by 75% MD containing roasted filler for 3 h. Both impact resistance and flexural strength decreased down by increasing roasting temperature from 900°C up to 1100°C. Mechanical properties improved again at the highest tested temperature by the formation of dicalcium silicates. Water absorption data drew similar proportional curve but in inverse direction. Change in the rate of free lime in the roasted filler explains the shoulder like trend curves of physical properties. Decrease in the rate of quartz was also thought to make contribution on drawing such curves. At lower temperatures, compactness of slab could not be improved due to the absence of calcium silicate sinter phases, incomplete calcination, and the presence of CaO phase at high rate (Gobechiya et al., 2008; Göktaş and Erdemoğlu, 2012; Kartal and Akpınar, 2004; Zadov et al., 2008).

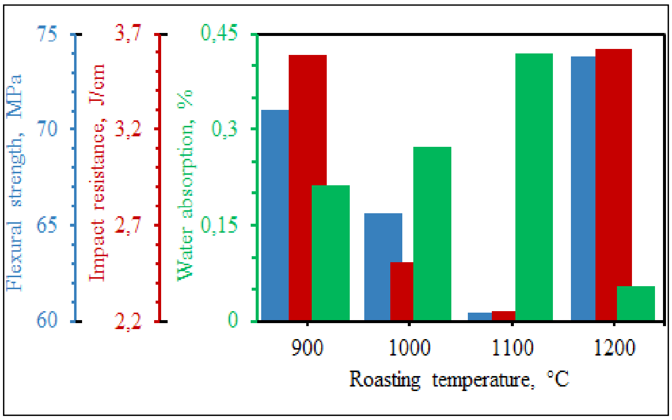

Figure 9. *Effect of roasting temperature on the mechanical and water absorption properties of composite slab prepared by roasted filler mixture containing 75% marble dust (roasting time: 3 h)*

Conclusions

Effect of marble dust (MD) containing roasted filler mixture on the physical properties of composite slab was investigated as economic and environmentally friendly approach. Experimental findings demonstrated promising results. MD was determined to have closer size distribution with micronized quartz filler. Resolution of XRD patterns exhibited that the roasted fillers contained wollastonite, larnite and calcio-olivine as Ca-silicate sinter phases in addition to free quartz and lime at changing ratios depending on the MD percentages, and roasting temperature and time. Mechanical properties of the slab prepared by filler mixture roasted at 900°C was measured to be closer to those of the reference quartz composite slab. As the roasting temperature was increased to 1100°C, physical properties retrogressed due mainly to the generation of free lime (CaO) at high rate. Conversely, improved physical properties were observed when the filler mixture was roasted at 1200°C. Better physical properties than those of reference quartz composite slab were obtained by roasting the 25% MD containing filler mixture for 10 h. Improved physical properties approaching to those of reference slab were also obtained at higher MD percentages when roasting was applied at least for 3 h. Comparative evaluation of phase distribution and physical properties demonstrated that larnite and calcio-olivine ameliorated the physical properties whereas free lime reduced product quality. High quality slab, even better than reference one, was produced by using 25% MD containing filler mixture roasted for 10 h. This result was attributed to the synergic effect of hard metastable larnite phase and quartz.

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