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Combined effect of pyrometallurgical and hydrometallurgical processes on quartz ore purification
Kuvars cevheri saflaştırılmasına pirometalurjik ve hidrometalurjik proseslerin bileşke etkisi

Ahmet Deniz Baş[a,*]

[a]*Muğla Sıtkı Koçman University, Faculty of Engineering, Mining Engineering Department, Muğla, TURKEY*

A B S T R A C T

Recent advances in high-tech applications have highlighted the growing demand on highly pure silicates like quartz. Therefore, purification of quartz ore was determined as the subject of this study performed by pyrometallurgical followed by hydrometallurgical processes. In this research, the effect of thermal treatment (TT) followed by oxalic acid (OA) bleaching of quartz was examined to have a better understanding on the relationship between Fe remaining in concentrate and colour response. The level of TT temperature was found to have a significant effect on the purification of quartz by OA. The maximum Fe rejection rate was observed to occur both for non-treated and TT quartz up to 250°C (Fe content decreased from 624 ppm to <100 ppm, and L* value increased from 81.34 to 88.23). TT between 400°C and 900°C showed poor purification performance: decreasing L* value, and increasing a* and b* values. It is important to note that further increase in TT temperature to 1100°C resulted in the poorest bleaching: Fe rejection rate decreased, but colour response improved providing the highest L* value and the lowest a* and b* values. This finding was explained by the formation of dissolution resistant iron silicates. Moreover, the rate of Fe removal from quartz ore and differences observed in its colour response by OA bleaching were explained by changes in crystalline structure and formation of microcracks.

Keywords: Quartz bleaching, Thermal treatment, Oxalic acid, Iron ion, Colour response.

Introduction

Highly pure silicates like quartz, SiO_2, and albite, Na-feldspar, are on high demand, and they are important raw materials in the manufacturing of high-tech products such as semiconductor microelectronics, optical devices, solar cells, nano applications in the production of paint, paper and plastics in addition to traditional ceramic applications (El-Rehiem an El-Rahman, 2008; Dong et al., 2020; Lin et al., 2020). The level of purity of silicates is a significant indicator that defines its usability in the production of advanced technology applications (Yang and Li, 2020). The presence of iron oxides as colouring impurities has negative effect as they lower the quality of final products by impairing transmission in optical fibers and the transparency of glasses, discolouring products and lowering the melting point of refractory materials (Taxiarchou et al., 1997; Müller et al., 2012; Vatalis et al., 2015). It is therefore essential to remove iron impurities associated with silicates (Zhang et al. 2012; Yang and Li, 2020). In order to remove/eliminate iron impurities from silicates, various approaches including physical and bio-/hydrometallurgical methods have been proposed by different research groups (Yan et al., 1987; Ubaldini et al., 1996; El-Rehiem and Abd El-Rahman, 2008; Sarvamangala and Natarajan, 2011; Zhang et al., 2012; Vapur et al., 2017).

Purification by physical methods often results in poor purity levels which is lower than the level needed in the advanced technology applications. Ghorbani and Haghi (2009) reported that the iron removal from feldspar by both wet magnetic separation alone and combined with flotation resulted in low performance with Fe content higher than 0.1%. Previous research studies on physical purification have shown that the first step is the removal of –25 μm fraction as slimes bearing clay minerals. The next step is the mica flotation at pH 2.5–3.5 (H_2SO_4) (Eddy et al., 1972; Bayraktar et al., 1998), where mica minerals are floated generally using long chain aliphatic amines (Baarson et al., 1962). After the removal of micaceous tailings, metal (Fe, Ti) oxides were floated either by oleate at pH 5–5.5 or by sulphonates at about pH 3–3.5. Besides the oleate and sulphonates, succinamates, soaps of various vegetable oils, sarcosine and hydroxamate type collectors can also be used for the flotation of metal-oxide minerals (Çelik et al., 1998; 2001). Yanjie et al., (2013) reported that the increase in magnetic field intensity has led to a decrease in concentrate yield and iron grade. The concentrate yield of total iron was found to be over 80% when the magnetic field intensity was 2.1 Tesla. Although high intensity permanent magnetic rolls are employed for the ores inherently low in iron, flotation is still indispensable for many ores especially the ones containing high

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amounts of titanium and iron (Orhan and Bayraktar, 2006). Mineral, e.g. feldspar, losses by physical methods is a significant issue in industrial practice which feldspar contant in tailings can reach up to 12% by weight (Orhan and Bayraktar, 2006).

Hydrometallurgical methods by dissolving the impurity minerals using various agents provide better results to overcome this issue (Ubaldini et al., 1996; Panias et al., 1996; Vegliò et al.,1999; Banza et al., 2006; Li et al., 2010; Zhang et al., 2012; Toncuk and akcil, 2016; Mohammed et al., 2019). Investigations have been made on the chemical purification using different inorganic and organic acids (Ubaldini et al., 1996; Vegliò et al., 1996; Banza et al., 2006; Zhang et al., 2012; Vapur et al., 2017). Among several organic acids, oxalic acid "OA" ($H_2C_2O_4$) is one of the most widely used in the dissolution of iron impurities. Iron oxide dissolution by OA can proceed via two main steps which are non-reductive and reductive dissolution (Bonney et al., 1996; Panias et al., 1996). The non-reductive dissolution refers to the presence of iron as Fe(III) oxalate complexes, while reductive dissolution involves an induction period describing the build-up of Fe(II) oxalate complexes in solution (Panias et al., 1996) Previous investigations have shown that OA has good complexation property, high reducing power, and can be recovered as by-product from other industrial practices (Taxiarchou et al., 1997; Lee et al., 2007; Du et al., 2011). Soluble complexes formed in OA bleach solution can easily be decomposed both microbiologically and under the action of daylight, thus allowing effective effluent treatment (Abbruzzese et al., 1999). OA was reported to provide the best bleaching performance over several different organic acids on the removal of iron from a kaolin sample (Ambikadevi and Lalithambika, 2000).

The dissolution of iron in OA results in the formation of bivalent and trivalent complex iron ions, $[Fe^{2+}(C_2O_4)_2]^{2-}$ and $[Fe^{3+}(C_2O_4)_3]^{3-}$, respectively. Free Fe^{2+} ion can be identified only in high acidic solutions, while free Fe^{3+} ion is not likely to build-up in OA solutions. In leach solutions with a pH range of 1-2, $[Fe^{3+}(C_2O_4)_2]^-$ and $[Fe^{3+}C_2O_4]^{2-}$ ions are stable, while $[Fe^{3+}HC_2O_4]^{2+}$ is the only complex ion exists at pH values lower than 1 (Cornell and Schindler, 1987; Litter et al., 1988; Panias et al., 1996; Torres et al., 1989). Du et al., (2011) obtained a 75.4% Fe removal by ultrasound effect during OA (4 g/L) bleaching of quartz at 95°C. Vegliò et al., (1998) observed very poor Fe removal performance, only a 3-9%, by using sulphuric acid alone at 90°C in drum reactor, but this was enhanced to 45% by mix of sulphuric acid (2 kg/t) and OA (3 kg/t) as reducing agent. The poor Fe removal was linked to the characterization of the sample as 52% of the iron was in the micaceous fraction, which was difficult to dissolve. In another study by Vegliò et al., (1999) 20-30% of iron removal was obtained when OA, 3 g/L, bleaching of quartz sand with an average size of 71 μm was performed at 80°C. Huang et al., (2013) reported an iron removal rate of 37.5% by OA at a concentration of 6 g/L. Taxiarchou et al., 1997; obtained a 40% of iron removal rate with the OA concentration of 4.5 g/L at 90-100°C at a particle size of 265 μm. These studies demonstrate the significance of bleaching agent type, leach temperature, and mineralogical properties of the sample in iron removal from quartz ore.

Different research groups have shown that the dissolution behaviour of iron is affected by the change in heat and acidity in the leach reactor (Panias et al., 1996; Lee et al., 2006; Patent 2014). In order to improve iron removal from quartz, heat treatment applications have also been tested (Loritsch and James, 1990; Li et al., 2021).Calcination is also a pretreatment technology in quartz process and widely combined with high temperature leaching for removing inclusion impurities within quartz. Crystal structures of some silicate minerals can be destroyed by calcination (Lin et al., 2018). Yang et al., (2018) examined the effect of calcination application at 900°C for 2 hours followed by mixed oxalic acid and hydrochloric acid bleaching of quartz. They reported a significant improvement in Fe_2O_3 content remaining in solid from 0.0857% to 0.0223%.

Colour response of quartz appears to be a significant indicator that determines the quality of product especially for its use in high-tech industries in addition to chemical leaching data, that is Fe removal from quartz. For industrial applications, white colour refers to quartz and calcite, red colour to hematite, and yellow colour to goethite (Vodyanitskii and Savichev, 2017). CIE-L*a*b* which is a universal colour space, and this is widely used to characterize colour under spectrometric analysis. The L* scale interprets the degree of whiteness in a range from 0 for pure black to 100 for diffuse white, positive value for scale "a" represents red colour, while negative value is the measure of greenness, the scale "b" shows yellowness for the positive value (+b) and blueness for negative (-b) (Green, 1999; Field, 2004). With the help of CIE-L*a*b*system, numerically a connection between the colour of quartz ore and contents of impurities in quartz can be established (Viscarra Rossel et al., 2006).In a recent study with oxalic acid, L* value was found to increase with increasing the iron rejection rate: the L* value increased from 80.02 to 88.70 at 0.04M OA, and reached to 89.23 at the 0.4M OA concentration. This finding was linked to the increased rejection rate of Fe colouring impurities was proportional to the increase in whiteness of quartz (Mohammed et al., 2019). Tuncuk and Akcil (2016) reported that sulphuric acid bleaching alone and in presence of oxalic acid yielded a 90.6 L* value.

In recent years, despite the fact that the industry still faces this issue, i.e. presence of high impurities, combined effect of pyrometallurgical and hydrometallurgical processes on quartz bleaching and its colour response has received little attentiton. The main objective of this study is to provide better understanding into thermal treatment (TT) at various temperatures on OA bleaching, and colour responses on the removal of iron from a quartz ore.

1. Materials and methods

The test material, quartz ore sample, at a particle size of -300+106 μm, was received from a quartz production facility in Yatağan region in Muğla, Turkey. Mineralogical characterization of the ore sample has shown that the sample is predominantly quartz together with hematite, magnetite, ilmenite and rutile as trace impurities (Mohammed et al., 2019). XRF and ICP analysis have shown that Fe_2O_3, TiO_2, MnO, SiO_2 contents of the feed sample were found to be 893 ppm, 419.4 ppm, 13.6 ppm and 87.2%, respectively, and this refers to 624.5 ppm Fe (Bas, 2021).

The washed test material, the feed, was first subjected to thermal treatment "TT" for 3 hours in muffle furnace (Ankatest) at various temperatures with a 10°C/min heating rate. At the end of TT, test material was left for cooling down to room temperature in the furnace, and then stored in nylon bags. Thermally treated test material was sampled as portions of 53 gram to be used in experiments to satisfy 10% solid by volume in the leach reactor. High purity analytical grade OA (Merck, Oxalic acid dihyrdate, assay ≥99.0%) was used as bleaching agent. The solution medium was prepared using distilled water. Agitated leaching was applied using a 500 mL glass reactor. Agitation at a rate of 400 rpm was supplied by a mechanical stirrer (M-TOPS MS-3020D) in which teflon coated impeller was used. Reactor was placed on hot plate to perform bleach tests. TT and OA bleaching tests conditions are presented in Table 1. TT was conducted at various temperatures for 180 minutes. Following TT, bleaching test was carried out in 0.1M OA solution at 90°C for 90 minutes. OA bleach test conditions were selected according to preliminary findings and the data available in literature (Mohammed et al., 2019).

Bleach reactor was closed to the atmosphere by using cooling tower and washing bottle. Leach solution was sampled during bleaching for Fe analyses at predetermined intervals. Over the test period, pH readings were noted. At the termination of bleaching test, bleached quartz (the concentrate) was washed thoroughly and then dried in oven at 105°C. Colour data, L*a*b*, was obtained by colorimeter from dried quartz product, which values for test sample were 81.81, 2.39 and 14.17 for L*, a* and b*, respectively. Metal analyses were performed on both feed, pregnant solutions, and bleached quartz by inductively coupled plasma mass spectrometry (ICP-MS). Samples after TT and after OA bleaching were characterized by optical microscopy (Motic, SMZ-140-N66).

Table 1. Experimental conditions with levels of parameters (0°C refers to the test without TT)

Exp. no	TT (°C)	TT Conditions	OA Bleach Conditions
1	0		
2	150		
3	250	180 min;	0.1M C2H2O4;
4	400	Heating rate:	90°C;
5	650	10°C/min	90 min
6	750		
7	900		
8	1100		

2. Results and discussion

2.1. Effect of TT on Fe Rejection Rate (%) by OA Bleaching from Quartz Ore Sample

The effect of TT temperature on Fe rejection rate (%) from quartz ore sample by OA bleaching is shown in Figure 1. Bleaching on the raw sample (without TT), Fe removal was 45% in the initial 15 minutes, and it reached to 72% at the end of the test period (90 minutes). TT of test samples at 150°C and 250°C resulted in 78% and 72% Fe rejection rates, respectively. Although Fe removal kinetics at 400°C was initially rapid (almost 50% Fe removal in the first 5 minutes), the kinetics slowed down for the rest of the test period. It is worth noting that TT temperatures tested above 400°C, i.e. 400°C-1100°C, the kinetics of Fe removal slowed down. This behaviour, i.e. slowdown in leach kinetics, at elevated temperatures over 400°C can be linked to the formation of more stable iron oxide phases in addition to appearance of iron silicates (Suarez et al., 2008).
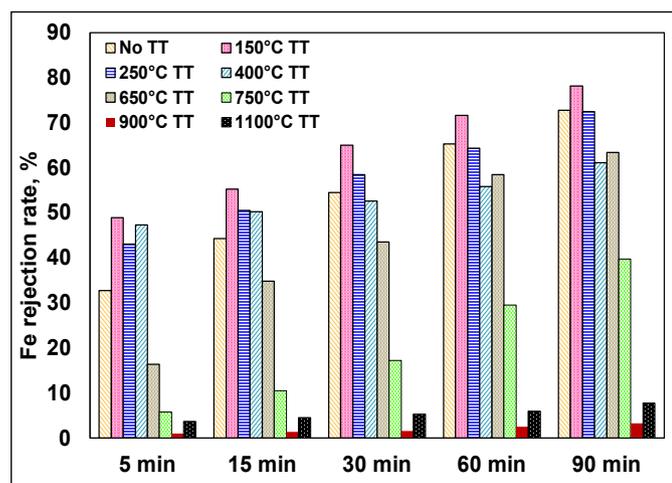
Figure 1. Effect of TT temperature for 3 hours on Fe removal kinetics from quartz by OA bleaching (0.1M OA; 90 °C; 90 min bleaching; 400 rpm)

Iron(III) oxalate complexes form in the solution during iron dissolution by OA. The trioxalatoferrate(III) ion [Fe(C₂O₄)₃]³⁻ is the most stable among iron(III) oxalate complexes. Thus, hematite (α-Fe₂O₃) dissolution can take place according to the reaction of Equation 1, which predominates in a solution at a pH higher than 4.0.

However, in a slightly acidic solution (pH 2.0 - 4.0), the complex ion [FeHC₂O₄]²⁺ can probably be formed in the solution, as described by the Equation 2 (Lee et al., 1997; Lee et al., 1999).

$$\alpha - Fe_2O_{3(s)} + 6C_2O_{4(aq)}^{2-} + 6H_{(aq)}^+ \rightarrow$$
$$2[(FeC_2O_4)_3]_{(aq)}^{3-} + 3H_2O_{(l)} \tag{1}$$

$$\alpha - Fe_2O_{3(s)} + 2HC_2O_{4(aq)}^- + 6H_{(aq)}^+ \rightarrow$$
$$2[(FeHC_2O_4)]_{(aq)}^{2+} + 3H_2O_{(l)} \tag{2}$$
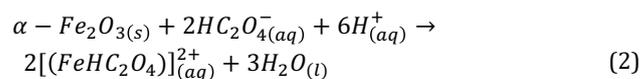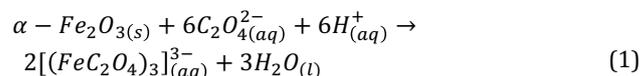
TT of quartz at high temperatures, e.g. 900°C, creates fractures which can lead to greater chemical activity during leaching. Hence, the impurities associated with quartz tend to react with bleaching agents along fractures. TT at elevated temperatures can promote quartz crystal distortion and then enhance leach kinetics of quartz. Therefore, high temperature TT is expected to help removing built-in inclusions in quartz ore (Lin et al., 2018)

2.2. Relationship Between Fe Remaining in Concentrate and Colour Response

When interpreting the whiteness index (WI), the "L*" value was taken into consideration as follows: a higher "L*" value symbolizes the whiteness of the material. To note that, Fe concentration and L*, a* and b* values in the feed sample (before bleaching) were found to be 624.5 ppm, 81.34, 1.5 and 11.4, respectively. In the absence of TT followed by OA bleaching of quartz, 98.8 ppm Fe remaining in concentrate and 88.01 of L* value were obtained (Figure 2). The lowest Fe remaining in concentrate of 95.2 ppm with 88.06 L* value was obtained when the sample was thermally treated at 150°C followed by OA bleaching. Fe remaining in concentrate and L* value lower than 100 ppm and 88.23, respectively, were obtained at temperatures up to 250°C.
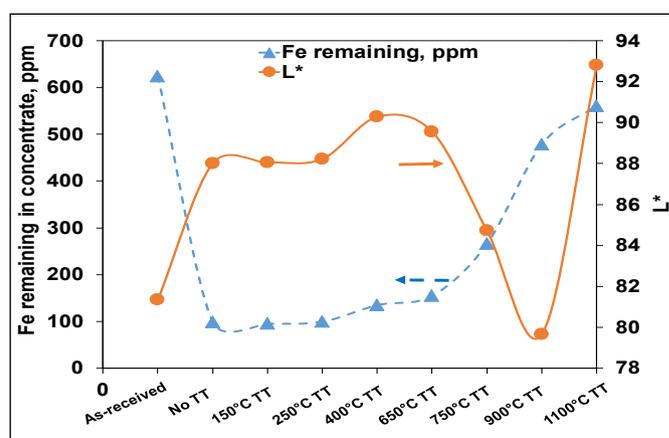
Figure 2. Relationship between Fe remaining in concentrate and colour response (TT at various temperatures for 180 min followed by 0.1 M OA concentration; 90 min bleaching; 90 °C, 400 rpm)

Further increase in TT temperature from 400°C up to 1100°C resulted in continuos decrease in Fe rejection rate, which corresponds to an increase in Fe remaining in bleached concentrate from 134.9 ppm to 560.9 ppm. However, L* value profile showed a differ-

ent trend as it slightly increased up to 90.29 at 400°C TT, decreased down to 79.67 at 900°C TT, and then reached to the highest L* value of 92.8 at 1100°C TT. In a study with sandstones, Gomez-Heras et al., (2008) observed first colour change at 250-300°C as a result of the commencement of thermal oxidation of iron-bearing minerals. It is important to note that the highest L* value resulted in unexpectedly the highest Fe remaining in concentrate. This can be explained by crack propagation as a result of phase change from α-quartz (2.67 g/cm³) to β-quartz (2.53 g/cm³) causing relative expansion in volume (Haja´l and Török, 2004).

In the case of Fe(III) oxide, the reduction of Fe^{3+} to Fe^{2+} helps enhancing the rate of dissolution. This enhancement can be linked to the great ability of Fe^{2+}-O bonds as compared to Fe^{3+}-O bonds (Baumgartner et al., 1983). The dissolution process involves reactions on the particle surface and linear dependence on $[C_2O_4^{2-}]$ that serves to a valence electron-transfer to Fe^{3+} ions on the surface as represented in Equation 3 (Baumgartner et al., 1983; Lee et al., 1997).

$$C_2O_4^{2-} + 2Fe^{3+} \rightarrow 2CO_2 + 2Fe^{2+} \qquad (3)$$
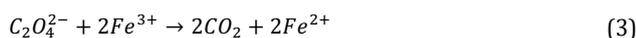
Experimental findings demonstrated that the relationship between Fe remaining in concentrate and colour response can be depicted in three zones (Figure 3). In Zone I (TT up to 250°C), a* and b* values, and Fe remaining in concentrate as average were found to be 0.75±0.11 and 3.45±0.20, and lower than 100 ppm, respectively. These results have revealed that TT up to 250°C showed almost no effect on quartz purification if compared to that of the bleaching of non-treated quartz ores sample. In Zone II (TT up to 900°C), a* and b* values and Fe remaining in concentrate increased by increasing TT temperature if compared to that of temperatures up to 250°C. The increase in Fe remaining in concetrate can be linked to the formation of dissolution-resistant phases. Kompaníková et al., (2014) observed increase in a* value of clay minerals after thermal treatment at temperatures above 400°C, which was linked to the iron oxidation and the transformation of clay minerals to mullite like phases.
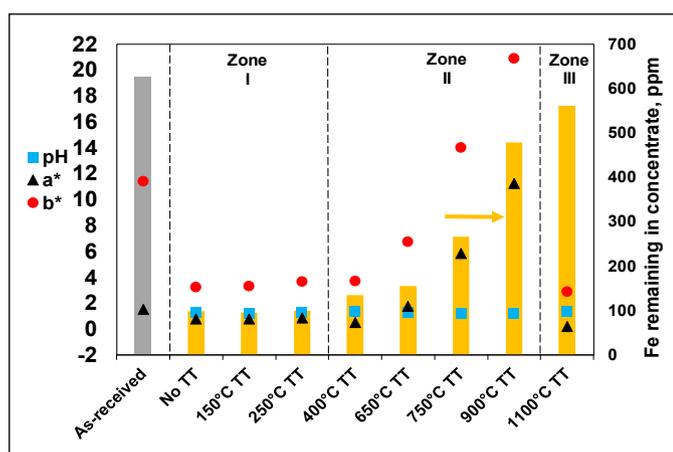
Figure 3. Relationship between Fe remaining in concentrate, pH and colour responses (TT at various temperatures for 180 min followed by 0.1 M OA concentration; 90 min bleaching; 90 °C, 400 rpm)

TT at 900°C followed by OA bleaching of quartz resulted in the highest a* and b* values, and the lowest L* value of 11.25 and 20.89, and 79.67, respectively. Phase change by increasing TT temperature from 250°C to 900°C resulted in the formation of firstly β-quartz (2.53 g/cm³) at temperatures of 580°C–595°C, and then to tridymite (2.30 g/cm³) (Haja´l and Török, 2004). Such a reasonable decrease

in solid density causes internal stresses in brittle quartz phase and creates numbers of cracks at grain boundaries mainly at temperatures above 600°C. Micro-cracks within the crystals appear mainly at higher temperatures above 750°C. For this quartz ore sample tested, the value of a* showed a positive shift from red to green with increasing temperature from 400°C to 900°C. The colour change is mostly related to the iron-bearing minerals. Therefore, significant decrease in the colour quality of quartz can be attributed to both formation of dissolution resistant phases and crack propagation (Haja´l and Török, 2004 Kompaníková et al., (2014) Vodyanitskii and Savichev, 2017). The presence of iron, as impurity, reduces the quality of quartz ore by decreasing brightness and increasing yellowness. Ferric (Fe^{3+}) impurities are known to bring an orange colour to quartz structure (Figure 4b). These findings have revealed that mineralogical deportment plays a key role on the leaching behaviour of colouring impurities by OA.
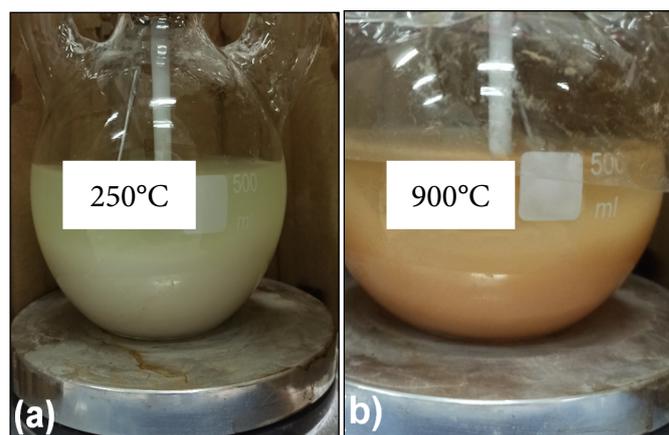
Figure 4. OA bleach liquor colour of thermally treated samples: (a) at 250°C; (b) at 900°C

In general, iron impairs the transparency of colourless container glass and high-quality glass, and transmission in optical fibers (Ubaldini et al., 1996; Vegliò et al.,1998). Further increase in TT from 900°C to 1100°C, i.e. zone III, demonstrated a significant improvement in colour response of quartz ore: reached to the highest L* value of 92.8 and to the lowest a* and b* values of 0.199 and 2.88. Although the sample post bleaching after TT at 1100°C had an improved whiteness index, this condition provided the lowest Fe rejection rates among all experiments, which corresponds to the highest Fe remaining in concentrate of 560.9 ppm. This behaviour at 1100°C can be explained by the formation of iron silicates (Suarez et al., 2008). The solution pH was reported to be a key parameter that controls the presence of various oxalate ions in bleaching system. The final pH values at the end of bleaching was found to have an effect on colour responses as function of TT temperature: the lowest pH of 1.18 was observed at 900°C, and the highest pH of 1.36 was obtained at 1100°C. Below pH 1.5, oxalic acid exists mainly as $H_2C_2O_4$, whereas HC_2O_4 is the most predominant species at pH 2.5–3.0. Final pH of leaching solution has been found to depend on the leaching time, initial pH for the leaching solution and the leaching temperature (Nwoye et al., 2020).

An important finding observed during bleaching tests is the difference in the colour of leach liquors. Bleaching of samples TT at 0°C (No TT), 150°C, 250°C, and 1100°C resulted in greenish colour (Figure 4.a), and the leach liquor had orange colour for samples TT at 400°C, 650°C, 750°C, and 900°C (Figure 4.b). The greenish and orange red colours can be attributed to the release of ferrous and ferric ions to the solution, respectively (Haja´l and Török, 2004).

Optimal microscopy images of the feed sample, and samples after TT and after OA bleaching are demonstrated in Figure 5. The representative images of bleached samples were selected from three different zones based on the findings presented in Figure 3. The brightest colour was observed after TT at 250°C followed by OA leaching (Figure 5.b). This indicator in whiteness was also suported by the increase in L* value from 81.81 to 88.23. Not only the L*value but also Fe remaining in concentrate was also found to be lower than 100 ppm at this condition (Figure 3). It is important to note that the solid sample colour became dirty orange-red colour after TT up to 900°C and OA bleaching. However, TT at 1100 °C resulted in a dirty white colour included with coloured black particles (Figure 5.e). Although it had the highest L* value at 1100°C, it showed the lowest Fe rejection rate which could be linked to the formation of iron silicates (Figure 2 and Figure 3) (Suarez et al., 2008).
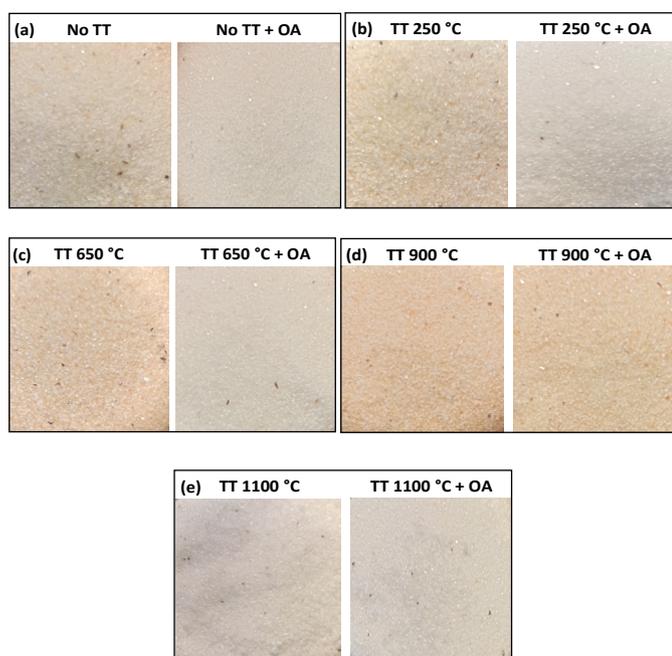
Figure 5. Optimal microscop images after TT at various temperatures and after TT + OA bleached samples, (a) TT 0 °C and OA; (b) TT 250 °C and OA; (c) TT 650 °C and OA; (d) TT 900 °C and OA; (e) TT 1100 °C and OA.

Conclusions

The effect of TT followed by acid bleaching of quartz was examined to have a better understanding on the relationship between Fe remaining in concentrate and colour response. The level of TT temperature on quartz bleaching was found to have a significant effect on the purification of quartz by OA. Fe remaining in bleached concentrate, and a* and b* values were found to decrease from 624 ppm down to 100 ppm, from 1.5 to 0.86, and from 11.4 to 3.65, respectively, and L* value increased from 81.34 to 88.23, when the sample was TT at temperatures up to 250°C. Increasing TT temperature from 400°C up to the maximum level tested showed a continuos increase in Fe grade remaining in concentrate. TT between 400°C and 900°C showed poor purification performance: decreasing L* value, and increasing a* and b* values. It is important to note that further increase in TT temperature, that is 1100°C, resulted in the poorest Fe rejection rate, but provided the highest L* value and lowest a* and b* values. Although colour response was significantly improved at 1100°C, it reported the poorest Fe rejection rate among other tests. The findings in this study suggest that changes in crystalline phase and formation of microcracks play important role on the removal of iron from quartz ore and colour response.

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