

Sonocatalytic treatment of baker's yeast effluent

E. Yılmaz and S. Findik

ABSTRACT

Baker's yeast effluent is a major source of pollution with a high organic load and dark colour. It can be treated by using advanced oxidation processes (AOPs). AOPs, such as ultrasonic irradiation, are ambient temperature processes involving the generation of free radicals. We have investigated sonocatalytic treatment of baker's yeast effluent by using ultrasound. $\text{TiO}_2\text{-ZnO}$ composites were used as sonocatalysts to increase the efficiency of the ultrasonic irradiation. The $\text{TiO}_2\text{-ZnO}$ composite was prepared by two different methods. Ultrasonic irradiation or mechanical stirring was used to prepare the $\text{TiO}_2\text{-ZnO}$ composite, and an ultrasonic homogenizer with a 20 kHz frequency was used to treat the baker's yeast effluent. We studied the effects of several parameters, including the molar ratio of $\text{TiO}_2\text{-ZnO}$, calcination temperature, calcination time and catalyst amount, on the sonocatalytic treatment of the effluent. According to the results, the decolorization rate was 25% when using the composite $\text{TiO}_2\text{-ZnO}$ prepared at a 4:1 molar ratio and treated at 700 °C for 60 min, and the optimum catalyst amount was 0.15 g/l.

Key words | baker's yeast effluent, decolorization, sonocatalyst, ultrasonic irradiation

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INTRODUCTION

Baker's yeast effluent is a major source of pollution as it has a high organic load and also has a dark color. The industrial production of baker's yeast by fermentation uses molasses as the raw material. Other industries that generate wastewater containing molasses include distilleries, fermentation industries and sugar mills. Molasses is the main by-product of sugar beet processing. The coloured compounds in molasses are known as melanoidins. Melanoidins are high molecular weight polymers and are formed during the reaction between amino compounds and carbohydrates. This reaction is known as the Maillard reaction. Melanoidins in wastewater prevent sunlight penetration, and reduce both photosynthetic activity and the dissolved oxygen level of the surface waters (Pena *et al.* 2003; Pala & Erden 2005; Liang *et al.* 2009a; Verma *et al.* 2011).

Biological treatment, generally a combination of aerobic and anaerobic processes, normally degrades melanoidins by

only 6 or 7% (Pena *et al.* 2003). Therefore, it is necessary to study additional treatments to remove colour from the molasses effluent and to prevent environmental problems. Various treatment methods like Fenton oxidation (Pala & Erden 2005), ozonation (Pena *et al.* 2003; Coca *et al.* 2005; Zeng *et al.* 2009), coagulation/flocculation (Zhou *et al.* 2008; Liang *et al.* 2009a, 2009b), electrocoagulation (Kobya & Delipinar 2008; Gengeç *et al.* 2012), ultraviolet oxidation (Çatalkaya & Şengül 2006; Dwyer *et al.* 2008), adsorption (Bernardo *et al.* 1997; Simaratanamongkol & Thiravetyan 2010) and ultrasound (Sangave & Pandit 2004, 2006; Sangave *et al.* 2007) have been tested to remove colour and chemical oxygen demand (COD).

The use of ultrasound for the treatment of wastewater has increased in recent years. Sonochemical oxidation uses ultrasound and results in cavitation phenomena, which consist of the formation, growth and collapse of cavities in an extremely small interval of time with simultaneous release of large amounts of energy. Water molecules dissociate into radicals due to the high localized temperatures and pressures. Pollutants in wastewater decompose because of the localized

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high concentration of species such as hydroxyl radicals and hydrogen peroxide in the solution (Sangave & Pandit 2004, 2006). However, in actual applications, the efficiency of using ultrasound alone to degrade organic compounds is relatively low. Many recent studies have focused on ultrasonic irradiation in combination with a photocatalyst (Wang *et al.* 2009; Abdullah & Ling 2010; Wang *et al.* 2010; Gao *et al.* 2011a, 2011b; Jamalluddin & Abdullah 2011; Anju *et al.* 2012; Ahmad *et al.* 2014).

Most studies on sonocatalytic degradation of pollutants in wastewater use TiO₂ as the catalyst, mainly due to its wide availability, the stability of its chemical structure, non toxicity, reactivity, high efficiency and low cost. ZnO, a similar semiconductor oxide, is reported to be more efficient than TiO₂ for visible light-induced photocatalytic degradation of organic pollutants (Gao *et al.* 2011a, 2011b; Anju *et al.* 2012).

However, no study on the ultrasonic treatment of baker's yeast effluent has been previously reported. Sangave & Pandit (2004, 2006) and Sangave *et al.* (2007) investigated ultrasonic treatment of distillery wastewater.

The purpose of this study is to remove colour and COD from baker's yeast effluent using ultrasound. A TiO₂/ZnO composite was used as a sonocatalyst. The composite was prepared using two different methods. Our experiments investigated the effects of the molar ratio of TiO₂/ZnO, the calcination temperature, the calcination time and the catalyst amount on the sonocatalytic decolorization and COD removal of baker's yeast effluent.

MATERIALS AND METHODS

Materials

The wastewater used in this study was obtained from a baker's yeast factory located in the northern part of Turkey. It was collected before biological treatment and

Table 1 | Characterization of baker's yeast wastewater

Parameter	Value
Absorbance at 400 nm	0.3–0.4
pH	5–6
COD	4,800–5,400 mg/l

stored at 4 °C. Table 1 shows the characteristics of the wastewater used in this study. The TiO₂ and ZnO were obtained from Merck. Distilled water was used throughout this study.

Apparatus

Ultrasonic irradiation was introduced using a probe type processor (HD2200; Bandelin, Berlin, Germany). Its operating frequency was 20 kHz and its power was 200 W. An ultrasonic bath (DSA50-SK-1; Fuzhou Desen Precision Instrument Co. Ltd, China) with a 42 kHz frequency and 1,600 ml volume was used for the preparation of the catalyst. A spectrophotometer (DR2400; Hach, USA) was used to measure the COD and absorbance of the samples. A COD reactor (Hach DRB 200) was used to heat the samples before measuring the COD value.

Preparation of TiO₂/ZnO composite

The TiO₂/ZnO composite was prepared by two different methods. The required amount of TiO₂ and ZnO to produce the required molar ratio (see results) was mixed, then distilled water was added until it covered the surface. Then the TiO₂/ZnO mixture was sonicated in an ultrasonic bath for 3, 6 or 12 min as required (see results) to improve the dispersion of TiO₂ and ZnO. After mixing was complete, the mixture was dried at 100 °C for 10 h and calcined at a specified temperatures (300, 500, 700 °C) and time (Wang *et al.* 2009, 2010). Alternatively, the TiO₂/ZnO was mixed by mechanical stirring, dried and calcined at 500 °C. For each method, the TiO₂/ZnO composite was prepared with various molar ratios.

Experimental procedures

A schematic diagram of the ultrasonic reaction system is shown in Figure 1. The wastewater was filtered to remove suspended particles using blue ribbon filter paper (Schleicher and Schuell). It was diluted with distilled water at the dilution ratio of 1:4 (wastewater volume/distilled water volume). The reactor used in this study was a 500-ml cylindrical glass vessel. The reactor was filled with 500 ml of diluted wastewater and the TiO₂/ZnO composite was added, then an ultrasonic probe was inserted into the

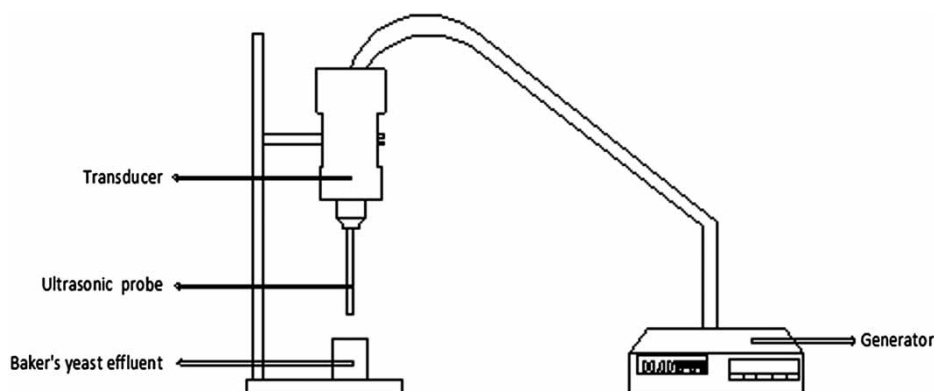


Figure 1 | Schematic diagram of the ultrasonic reaction system.

reactor. The probe was positioned 3 cm above the bottom of the reactor. The power of the ultrasonic probe was adjusted to 80 W and pulsed at a cycle of 30%. An experiment took 60 min, and all the experiments were repeated at least three times. Samples were taken from the reaction mixture periodically and centrifuged at 4,000 rpm for 10 min to remove any suspended particles and catalyst. After that, the absorbance and the COD of the sample were recorded using a spectrophotometer.

An absorbance measurement was done at different wavelengths to specify the maximum absorbance value. The absorbance of the effluent was measured at 400 nm wavelength, as maximum absorbance was obtained at this wavelength. The absorbance of the effluent was evaluated to calculate decolorization.

COD was measured using the DR 2400 spectrophotometer following the instructions for the Hach higher range test. To measure COD, a 2 ml sample was placed into a special vial containing a dichromate solution, then it was heated in the DRB 200 COD reactor at the desired temperature and time. After cooling the vial, the COD of the sample was recorded using a spectrophotometer.

Reaction temperature was not controlled in any of our experiments except those intended to investigate the effect of temperature. No pH adjustment was done during the sonication, hence the pH of the effluent was the same as the initial (pH 5–6) for the treatment.

The effect of temperature was investigated in the absence of the TiO_2/ZnO composite. Experimental conditions for the sonocatalytic treatment of baker's yeast effluent using the TiO_2/ZnO composite are given in Table 2. The effect of the

composite preparation method and composite molar ratio were investigated. The effect of the ultrasonic irradiation time for catalyst preparation was investigated using a 4:1 molar ratio. Then, the effect of calcination temperature and calcination time were studied. Finally, the effect of the TiO_2/ZnO composite quantity was investigated by using composite calcined at 700 °C for 60 min.

RESULTS AND DISCUSSION

The effect of solution temperature on sonolytic treatment of baker's yeast effluent

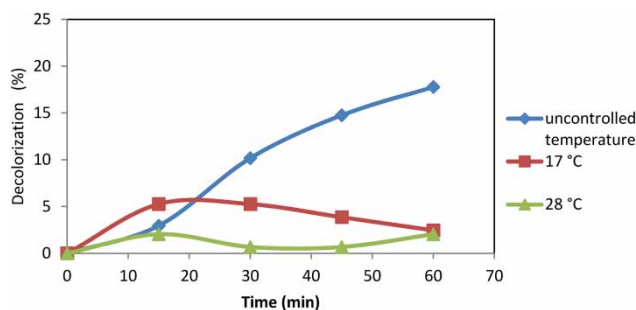
The temperature effect was investigated at 17 °C, 28 °C and with an uncontrolled temperature. To supply a constant temperature, a reaction vessel with a cooling jacket was used.

In the present study, an uncontrolled temperature means there is no external cooling mechanism to control the bulk solution temperature in the reactor. Due to the dissipation of ultrasonic energy in the liquid, the temperature of the reaction mixture increases gradually as the ultrasonic irradiation time increases. An increase in the bulk solution temperature indicates an increase in energy entering the reactor. In this study, the experiments started at an ambient temperature of about 20 °C and reached 40 °C at the end of the first hour. Similar results have been reported for the ultrasonic treatment of distillery wastewater (Sangave & Pandit 2004, 2006).

Figure 2 shows the effect of solution temperature on the sonolytic decolorization of baker's yeast effluent: there is no

Table 2 | Experimental conditions used in this study

Run	TiO ₂ /ZnO composite preparation method	Ultrasonic irradiation time/mechanical stirring time (min)	TiO ₂ /ZnO composite molar ratio	Calcination temperature (°C)	Calcination time (min)	Amount of TiO ₂ /ZnO composite (g/l)
1	Ultrasonic irradiation	6	1:0	500	60	0.2
2	Ultrasonic irradiation	6	4:1	500	60	0.2
3	Ultrasonic irradiation	6	3:2	500	60	0.2
4	Ultrasonic irradiation	6	2:3	500	60	0.2
5	Mechanical stirring	6	1:0	500	60	0.2
6	Mechanical stirring	6	4:1	500	60	0.2
7	Mechanical stirring	6	3:2	500	60	0.2
8	Mechanical stirring	6	2:3	500	60	0.2
9	Ultrasonic irradiation	3	4:1	500	60	0.2
10	Ultrasonic irradiation	12	4:1	500	60	0.2
11	Ultrasonic irradiation	6	4:1	300	60	0.2
12	Ultrasonic irradiation	6	4:1	700	60	0.2
13	Ultrasonic irradiation	6	4:1	700	30	0.2
14	Ultrasonic irradiation	6	4:1	700	90	0.2
15	Ultrasonic irradiation	6	4:1	700	60	0.1
16	Ultrasonic irradiation	6	4:1	700	60	0.15

**Figure 2** | Effect of the solution temperature on the sonolytic decolorization of the baker's yeast effluent.

positive effect of controlling temperature, so other experiments were done at an uncontrolled temperature. If the increase in temperature is beneficial for decolorization, the cooling water would not be needed, so the operation cost for cooling water would be saved (Zhang *et al.* 2006). According to Sangave & Pandit (2004, 2006), bulk solution temperature affects the formation of different intermediate products due to the complex structure of wastewater coupled with the acoustic effects of ultrasound. A chemical transformation takes place owing to acoustic cavitation. Chemical bonds are broken due to energy inside the reactor.

Scanning electron microscopy and surface analysis of TiO₂/ZnO composite

Typical scanning electron microscopy (SEM) images of TiO₂/ZnO (molar ratio 4:1) are shown in Figure 3. The surface appearance of the composite TiO₂/ZnO prepared by stirring (Figure 3(a)) or in an ultrasonic bath (Figure 3(c)) is shown. In the composite prepared using ultrasound, agglomeration of the particles was observed and the porosity increased. This observation was consistent with the surface analysis results. As shown in Table 3, the Brunauer–Emmett–Teller (BET) surface area, the pore volume and the pore diameter increased with ultrasonic preparation.

Figure 3(b)–3(d) show SEM images of the composite TiO₂/ZnO prepared with ultrasonic irradiation at 300 °C, 500 °C and 700 °C for 60 min. The composites consist of spherical particles and many mesopores. As the calcination temperature increased, the particles aggregated to form larger particles. A significant agglomeration between the spherical particles was observed when the calcination temperature increased from 300 to 500 °C, consistent with the surface analysis results. As shown in Table 3, the BET surface area increases as calcination temperature increases from 300

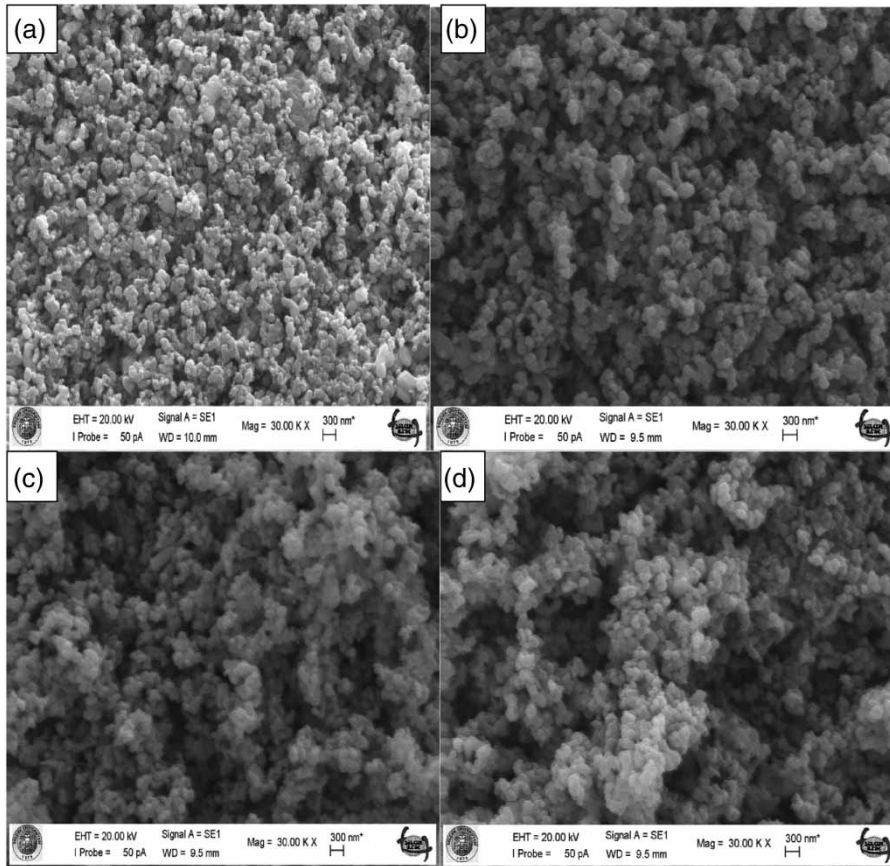


Figure 3 | SEM images of the composite TiO_2/ZnO (molar ratio 4:1, calcination time 60 min): (a) prepared with mechanical stirring, calcination temperature 500 °C, (b) prepared with ultrasonic irradiation, calcination temperature 300 °C, (c) prepared with ultrasonic irradiation, calcination temperature 500 °C, (d) prepared with ultrasonic irradiation, calcination temperature 700 °C.

Table 3 | Surface analysis of the prepared composites TiO_2/ZnO at different conditions

Composite preparation method	Calcination temperature (°C)	BET surface area (m^2/g)	Pore volume (cm^3/g)	Pore diameter (nm)
Mechanical stirring	500	8.2707	0.013612	5.9881
Ultrasonic irradiation	300	8.4809	0.014139	6.1972
Ultrasonic irradiation	500	8.6457	0.014508	6.0630
Ultrasonic irradiation	700	8.6141	0.013036	5.7960

to 500 °C. A reduction in the BET surface area from 8.6457 to 8.6141 m^2/g resulted when the calcination temperature was increased from 500 to 700 °C. The pore diameter and pore volume of the particles also decreased at 700 °C. At a high

calcination temperature, a reduction in the BET surface area and pore diameter may be the result of phase transformation, crystal growth and the collapse of the mesoporous structure (Jamalluddin & Abdullah 2011).

The effect of molar ratio of the composite TiO_2/ZnO on sonocatalytic decolorization of baker's yeast effluent

Sonocatalytic degradation of baker's yeast wastewater in the absence of a catalyst was first studied by ultrasonic irradiation. Without the catalyst, the decolorization was about 17% within a 60 min period. The presence of a catalyst can enhance the dissociation reaction of H_2O molecules to increase the number of free radicals generated (Pang *et al.* 2011).

Experiments were also performed to test adsorption. The reactor was filled with 1:4 diluted wastewater and the chosen amount of TiO_2/ZnO composite, and magnetically

stirred at an ambient temperature and at a constant temperature of 40 °C. Samples were withdrawn periodically to test decolorization and removal of the COD. There was no change in the colour and COD. These results showed that there was no adsorption with stirring.

Figure 4 shows the removal of colour by sonocatalytic treatment in the presence of TiO₂/ZnO composite prepared either by treatment in an ultrasonic bath or by mechanical stirring. There was no response for the composite TiO₂/ZnO prepared with mechanical stirring except for the 4:1 molar ratio; also there was no response for the composite prepared at the 2:3 molar ratio by either method. The composite prepared with ultrasonic irradiation at a 4:1 molar ratio and calcined at 500 °C for 60 min gives the largest decolorization. An agglomeration of particles and the formation of mesopores may affect the production of OH radicals. The BET surface area increased from 8.2707 to 8.4809 m²/g for the composite prepared by ultrasonic irradiation. The catalytic activity increases with an increase in surface area. High surface area provides more active sites for the production of free radicals (Jamalluddin & Abdullah 2011). According to Wang *et al.* (2009), the ultrasonic irradiation applied during the catalyst preparation improves the dispersion of the TiO₂ and ZnO.

The effect of ultrasonic irradiation time in the preparation of the composite TiO₂/ZnO on sonocatalytic decolorization of baker's yeast effluent

To investigate the effect of ultrasonic irradiation time in the preparation of composite TiO₂/ZnO, the catalyst was

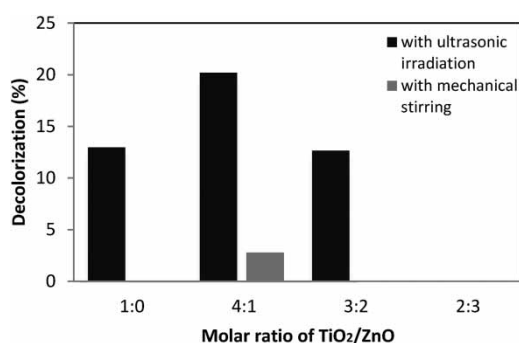


Figure 4 | Effect of the molar ratio of TiO₂/ZnO prepared with ultrasonic irradiation/mechanical stirring on sonocatalytic decolorization of baker's yeast effluent within 60 min (catalyst amount 0.2 g/l, calcination temperature 500 °C, calcination time 60 min, ultrasonic irradiation/mechanical stirring time 6 min).

prepared at a 4:1 molar ratio with 3, 6 or 12 min irradiation time. The use of ultrasonic irradiation during the preparation of catalyst improves the dispersion of two oxides. Ultrasonic irradiation time for preparation of the composite may effect interaction of the oxides. As shown in Figure 5, 6 min was the optimum irradiation time for the preparation of the composite. Interaction between TiO₂ and ZnO particles may be highest when the ultrasonic irradiation time is 6 min.

The effect of calcination temperature of the composite TiO₂/ZnO on sonocatalytic decolorization of baker's yeast effluent

The sonocatalytic activity of TiO₂/ZnO (4:1) calcined at different temperatures is shown in Figure 6. The activity of the composite increases with increasing calcination temperature. A possible explanation is that the interaction between TiO₂ and ZnO is strongest at 700 °C. The BET surface area increased from 8.4809 to 8.6457 m²/g when the calcination temperature rose from 300 to 500 °C then decreased to 8.6141 m²/g at 700 °C. The high surface area provides more surface area for active sites to produce ·OH radicals. The large surface area of the catalyst might be an important factor for sonocatalytic reactions, but it is not the main contributor in obtaining the highest sonocatalytic activity. The high surface area is just a requirement, but not a crucial factor. In a study of a combined Fe (III)/TiO₂ catalyst, the balance between the BET surface area, the proper amount of particles and the crystal phase was a major important factor in determining the sonocatalytic activity (Jamalluddin & Abdullah 2011).

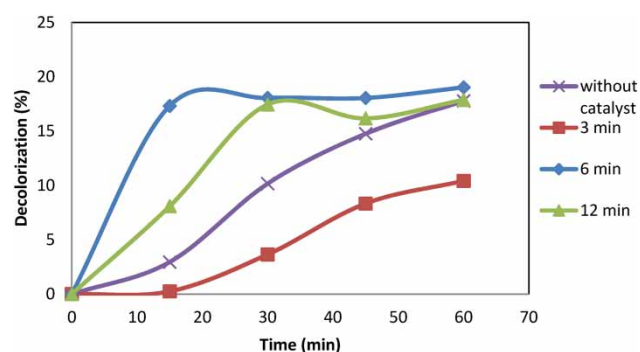


Figure 5 | Effect of ultrasonic irradiation time for the preparation of TiO₂/ZnO catalyst with a 4:1 molar ratio on sonocatalytic decolorization of baker's yeast effluent (calcination temperature 500 °C, calcination time 60 min, catalyst amount 0.2 g/l).

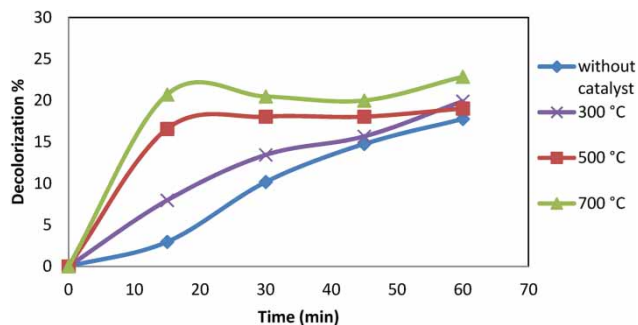


Figure 6 | Effect of the calcination temperature on the sonocatalytic decolorization of the baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination time 60 min, catalyst amount 0.2 g/l).

The effect of calcination time of the composite TiO_2/ZnO on sonocatalytic decolorization of baker's yeast effluent

The effect of calcination time was investigated at a calcination temperature of 700 °C. As shown in Figure 7, the decolorization is highest for a calcination time of 60 min. The decolorization efficiencies are 11%, 24% and 21% for the heat treatment, for 30 min, 60 min and 90 min, respectively. Below 60 min calcination time, interaction between the particles may be weak and above 60 min calcination time, a small amount of inactive phases are formed that inhibit decolorization. A similar result was obtained by Wang *et al.* (2009), who investigated the influence of heat treatment time on the sonocatalytic activity of composite TiO_2/ZnO powder at 10 min intervals, from 20 min to 60 min. According to their results, the best degradation ratio was obtained with a heat treatment of 50 min. For the short heat treatment, the composite TiO_2/ZnO can not be well activated. For the long heat treatment, sonocatalytic activity

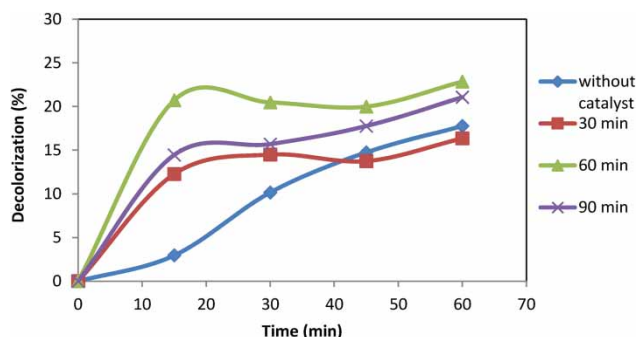


Figure 7 | Effect of the calcination time of the composite TiO_2/ZnO on the sonocatalytic decolorization of the baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination temperature 700 °C, catalyst amount 0.2 g/l).

decreases due to the transformation of TiO_2 from the anatase to rutile phase, and the existence of ZnTiO_3 impurity. Gao *et al.* (2011a) observed similar results in an investigation of the heat treatment time (30, 60 and 90 min) on the sonocatalytic activity of the $\text{Er}^{3+}:\text{YAlO}_3/\text{TiO}_2\text{-ZnO}$ composite. According to their results, the 60 min heat treatment degradation ratio has a maximum value. Below 60 min, the degradation ratio is low. According to their study, the components treatment at 30 min are not tightly bound. When the heat treatment continues for 90 min the partly Zn^{2+} composite began to enter the TiO_2 lattice and a tiny amount of inactive phase is formed which inhibits the degradation of acid red B.

The effect of the composite TiO_2/ZnO dosage on sonocatalytic decolorization of baker's yeast effluent

Experiments were done on different amounts of TiO_2/ZnO with a 4:1 molar ratio, a calcination temperature of 700 °C and a calcination time of 60 min. As shown in Figure 8, 0.15 g/l catalyst gives the highest decolorization. As the catalyst amount increases from 0.15 to 0.2 g/l, the decolorization decreases. An increase in the catalyst amount provides an increase in the total surface area of the catalyst, so the generation of OH radicals intensifies. On the other hand, an excessive amount of catalyst may inhibit the dissipation of ultrasound and the generation of radicals, so sonocatalytic activity decreases.

Similar results were obtained by Jamalluddin & Abdullah (2011) and Abdullah & Ling (2010). The lower removal rate at high loads could be explained by the fact that too

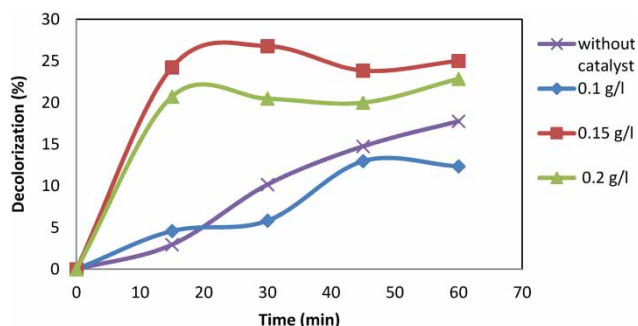


Figure 8 | Effect of catalyst amount on sonocatalytic decolorization of baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination temperature 700 °C, calcination time 60 min).

much catalyst added into a reaction system could cause a mutual screening effect among the catalyst particles. Thus, the energy provided by the ultrasound could not reach the surface of the catalyst and consequently resulted in a lower generation of active radicals.

COD removal

The samples that were withdrawn were analysed for changes in the COD. The effect of the ultrasound on the COD removal from the baker's yeast effluent is shown in Figure 9. There is no reduction in the COD of the effluent sample due to the passage of ultrasound in any treatment. In this study, real wastewater containing coloured compounds (melanoidins) was used. The melanoidins produce various intermediate products with ultrasonic irradiation. Due to the formation of a different intermediate transformation product, there is no change in the COD value.

There are similar results in the literature. For example, Sangave & Pandit (2004, 2006) obtained similar results for the distillery wastewater. In the case of real wastewater systems, ultrasonic irradiation leads to the formation of multitudes of products due to the inherent complexity of the effluent which in turn are difficult to identify. The low frequency of ultrasound caused the disintegration of the pollutant molecules in to smaller fractions rather than their complete reaction. In another study (Dükkancı & Gündüz 2013), there was no COD reduction for the sonocatalytic degradation of butyric acid in aqueous solutions. According to the results, butyric acid could be oxidized into lower molecular weight carboxylic acids rather than the end product of the CO_2 .

CONCLUSIONS

We have studied sonocatalytic decolorization and the COD removal of baker's yeast effluent. We investigated the effect of several parameters, such as the composite preparation method, the molar ratio of TiO_2/ZnO , the calcination temperature and time and the catalyst amount, on the sonocatalytic treatment of the baker's yeast effluent. It was found that the decolorization of the baker's yeast effluent

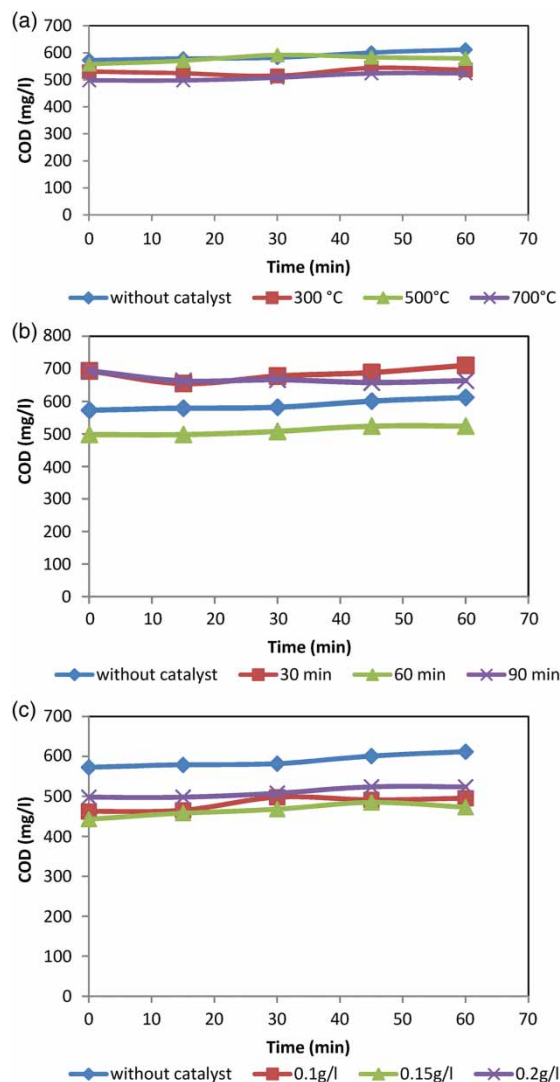


Figure 9 | (a) Effect of calcination temperature on sonocatalytic COD removal of baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination time 60 min, catalyst amount 0.2 g/l). (b) Effect of calcination time on the sonocatalytic COD removal of baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination temperature 700 °C, catalyst amount 0.2 g/l). (c) Effect of the catalyst amount on the sonocatalytic COD removal of the baker's yeast effluent (molar ratio of TiO_2/ZnO 4:1, calcination temperature 700 °C, calcination time 60 min).

was higher at a 4:1 molar ratio of the TiO_2/ZnO composite prepared with ultrasonic irradiation. The decolorization increased with an increase in the calcination temperature, and 60 min is the optimum calcination time. The catalyst amount which gave maximum decolorization was 0.15 g/l. There is no COD removal in the studied conditions. The combination of ultrasound with other advanced oxidation processes such as ozonation, UV, or photolysis can give better results than ultrasound alone.

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