

Synthesis of ZnO nanoparticles for microwave-induced rapid catalytic decolorization of congo red dye

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ABSTRACT

The ZnO nanoparticles (ZnO NPs) were synthesized by co-precipitating method followed by calcining at 500°C for 4 h. The characterization data show that the synthesized ZnO NPs were present in hexagonal wurtzite structure and space group P63mc with high purity and good crystalline nature. The synthesized ZnO NPs were tested for decolorization of congo red (CR) dye under microwave irradiation using microwave induced catalytic (MIC) method. The investigation results showed that the microwave induced ZnO NPs process could effectively decolorize the CR dye with the efficiency approaches 90.63% in a short contact time under given condition of 10 mg/l CR dye, 300 watt of microwave power, 3 min of contact time, and 3 g/l of catalyst dosage. Since the microwave-induced catalytic process in this study is noticeable, the microwave technology coupled with ZnO NPs as catalyst may have synergistic effects on the decolorization of CR dye giving high degradation ratio in short reaction time. Therefore, the microwave technology can be used as an alternative option to enhance the catalytic activity of metal nanoparticles dealing with removal of various industrial dyes containing wastewater. Copyright © 2013 VBRI press.

Keywords: Zinc oxide; nanoparticle; microwave; catalytic; decolorization; congo red; dye.



Oman Zuas is working as a researcher in Process Technology and Catalysis (PTC) Division, Research Centre for Chemistry, Indonesian Institute of Sciences (RCC-LIPI). His research interests include develop of metal oxide-based and agriculture waste materials, and their use as adsorbent for the removal of environmental pollutants. He is also engaged in the scientific research in designing and developing infrastructure for Gas Metrology, and preparing the Certified Reference Material (CRM) for gas mixtures.



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Introduction

Continually release of dyes containing wastewater into water bodies is growing a global pollution problem as a result of their wide industrial uses. In aquatic environment, the dyes undergo biochemical changes in aquatic environment and consume dissolved oxygen, which further threat to the living organism in aquatic ecosystem, as well as human health. Consequently, it is definitely necessary to remove the dye contaminant before discharging them into streams. In this regards, catalytic oxidation treatment is a great potential technology and the results of its application have been reported in published literatures [1-6]. Despite the fact that the aims to improve the removal efficiency of toxic dye from industrial wastewater using catalytic oxidation method have been pointedly achieved, a method development in the area is still facing challenges. Likely, one of the promising ways is by combining the use of material-based solid catalysis with microwave technology, so called microwave induced catalytic (MIC) method. The microwave technology for dye removal give an advantage effect because microwave irradiation may induce dipole orientation and migration of polar molecule of substances (such as water) in the reaction mixture which lead the

molecules to higher-excited state and further increase the collision numbers between reactants in the reaction mixture [7]. Hence, combination of microwave heating and catalyst would generate a synergism effect that can accelerate the rate of catalytic reaction in comparison with a microwave heating or the catalyst alone. Zhang *et al* [8] reported that a high degradation ratio in a short reaction time can be achieved for catalytic degradation of congo red in the presence of activated carbon under microwave irradiation assistance. He, *et al* [9] studied the use of nano nickel dioxide catalyst for crystal violet dye removal from aqueous suspension. They found that 97% of a 100 mg/l crystal violet can be rapidly degraded in 5 min. Yuan *et al* [10] observed that 50 mg/l reactive brilliant blue suspension can be rapidly decolorized in 8 min when MnO/MgO was used as catalyst under assistance of microwave irradiation. Other investigation on removal of dyes using MIC method such as methyl orange [11], Argazol Blue BF [12], remazol golden yellow [13], and acid indigo [14] in aqueous solution have also been reported in published literature.

In the last decade, the nanostructured of metal oxides have attracted considerable interest in both academic research and industrial applications. Among the metal oxides, a special attention has been devoted to nanostructured ZnO. The ZnO is a versatile semiconductor because of its wide application in the various research fields such as solar cell, panel display, photocatalysis, and electronic, environmental, and biomedical [15]. For energy generation, the nanostructured ZnO has been reported to be a promising material for increasing the photo conversion efficiency in solar cell application [16]. The ZnO is also found for possible use as transistor electronic material in an active-matrix liquid-crystal display [17]. In the field of environmental protection, the nanostructured ZnO can be used as an active material to treat water containing organic contaminants and inactive bacteria and viruses [18]. Recently, biomedical application of the nanostructured ZnO has been investigated, showing that the nanostructured ZnO is very useful as anti bacterial agent to treat pathogenic bacteria [15].

As was previously mentioned, some active catalysts have been used to remove dye from aqueous solution under the assistance of microwave irradiation. The strong activity obtained was not only due to the activity of the catalyst itself, but also the microwave irradiation used has contributed to the enhancement of the catalytic activity rate by increasing the collision numbers between reactants in the reaction mixture. In this regards, we assumed that the synergistic effect between the ZnO NPs and microwave irradiation may serve better capacity to remove the dyes from aqueous solution than the ZnO NPs alone without microwave irradiation.

In this study, the specific aims was to investigate the possible application of the synthesized and characterized ZnO NPs for decolorization of aqueous solution containing CR dye using MIC method. The effect of reaction parameters on the efficiency of the MIC process including initial concentration of CR dye, ZnO NPs catalyst dosage, and contact time were studied. For comparison, the decolorization testing under different treatment processes were also investigated.

Experimental

Reagents

Ammonia solution (NH₄OH, 25%, Merck-Germany), N-Cetyl-N,N,N-trimethylammonium bromide (CTAB, 98%, Merck-Germany), Zinc chloride (ZnCl₂, 98%, Merck-Germany), and Zinc oxide (ZnO, specific surface area 47.2 m² g⁻¹; average primary particle size 230 nm, Merck-Germany) and Congo red dye (C.I. 22120, 97%, Sigma-Aldrich, Germany) were used as received without further purification. Purified water was produced from a NanoPure purification system (Millipore Corp., 17.5 MΩ·cm) and was used throughout experiments.

Synthesis of ZnO NPs

The synthesis procedure of ZnO NPs was done via co-precipitation method [19, 20]. In a typical experiment, a certain amount of ZnCl₂ was dissolved in a 0.2 M CTAB solution under vigorously stirring for 5 min, giving a solution-A. The solution-A was then precipitated using NH₄OH solution with 5 ml/min of dropping rate under stirring until the pH value of the solution was 10 (solution-B). The solution-B was aged at 55-60°C under stirring for 6 h, giving a solution-C. The solution-C was then centrifuged at 8000 rpm for 10 min for several times until no Cl⁻ ions in the supernatant could be detected. The wet solid from centrifugation was dried at 100°C for 12 h to produce dry solid precursor. The synthesized ZnO NPs were obtained by calcining the dry solid precursor at 500°C for 4 h.

Characterization of the synthesized ZnO NPs

The synthesized ZnO NPs were characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and transmission electron microscopy (TEM). The XRD pattern was recorded on a Philips Analytical PW3710 X-ray diffractometer operated at 40 kV and 100 mA with CuKα as the radiation source. The 2θ was scanned in the range of 25 to 75 at speed of 1.2 min⁻¹. The FTIR spectra were recorded on a Shimadzu IR Prestige DSR-8000 spectrometer in KBr media in the range 400 to 4000 cm⁻¹ at a scanning rate 4 cm⁻¹/min. The morphological structure and EDAX were conducted on a JEM-2100F high resolution transmission electron microscopy (HRTEM) operating at 200 kV. The TEM sample was dispersed in ethanol, sonicated and then one drop of the suspensions was deposited onto a 300 mesh copper grid.

Catalytic evaluation of the synthesized ZnO NPs

The microwave-induced rapid catalytic decolorization (thermed as MIC) was applied to evaluate the activity of the synthesized ZnO NPs. The MIC experiments were carried out using a thermostatic microwave apparatus (Milestone Microwave Laboratory System, MLS 1200) by the following procedure. CR dye solutions (10 ml) with known initial concentration were transferred into a teflon vessel followed by adding a certain dosage of the synthesized ZnO NPs. Subsequently, the filled vessel was put into the microwave apparatus and the catalytic experiment was started. After that, the treated CR dye suspension was taken out from the microwave apparatus and was then centrifuged at 2200 rpm for 5 minute to separate the solution from the

ZnO NPs. The absorption of the treated CR dye solution was measured using a Shimadzu 3501 UV-Vis spectrophotometer at maximum wavelength of 497 nm. The remaining concentration of the treated CR dye solution was calculated from a calibration curve ($A = 0.0724C + 0.0131$) with $R^2 = 0.9989$, which was obtained from the maximal absorbancies of a series of CR dye solutions with concentration ranged from 0 to 10 mg/l). The calibration curve, which abides to Lambert-Beer's law, was used to determine the CR dye concentration in the treated solution. The efficiency of the synthesized ZnO NPs to decolorize the CR dye is expressed as follows:

$$\text{Decolorization efficiency (\%)} = \frac{(C_o - C_t)}{C_o} \times 100$$

where C_o (mg/l) and C_t (mg/l) are the CR dye concentration at initial and after time t (min), respectively.

Results and discussion

Characterization

Fig. 1 presents the XRD pattern of the synthesized ZnO NPs. It can obviously observed from the **Fig. 1** that the synthesized ZnO NPs have well-defined diffraction peaks at about 31.81° , 34.45° , 36.29° , 47.57° , 56.63° , 62.89° , 66.43° , 67.95° , 69.07° , and 72.69° that are assigned to the (100), (002), (101), (102), (110), (103), (200), (112), (201), and (004), respectively. This XRD pattern is in good agreement with crystal planes of ZnO having both hexagonal wurtzite structure and space group P63mc (JCPDS card no. 36-1451) [21]. The XRD pattern also exhibits sharp diffraction peaks, indicating that the synthesized ZnO NPs were presence in high crystal quality. The average crystallite size of the synthesized ZnO NPs was calculated from the full-width at half-maximum (FWHM) of (101) diffraction peak using Scherrer's formula [22], $D = k\lambda / \beta \cos \theta$, where D is the crystallite size, k is a constant ($= 0.9$ assuming that the particles are spherical), λ is the wavelength of the X-ray diffraction, β is the FWHM, θ is the angle of diffraction. It was found that the synthesized ZnO sample had calculated average crystallite size of ca. 10.5 nm.

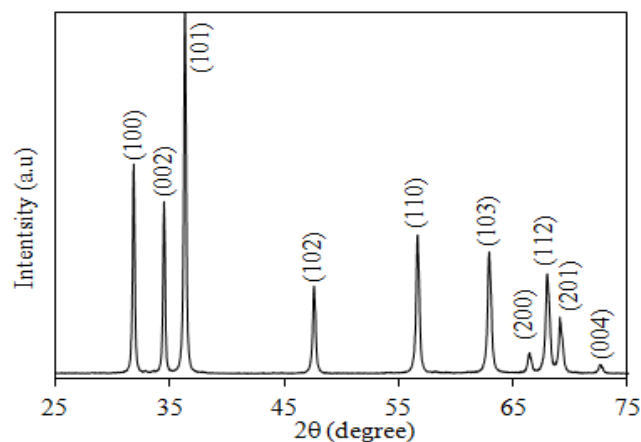


Fig. 1. The XRD pattern of the synthesized ZnO NPs.

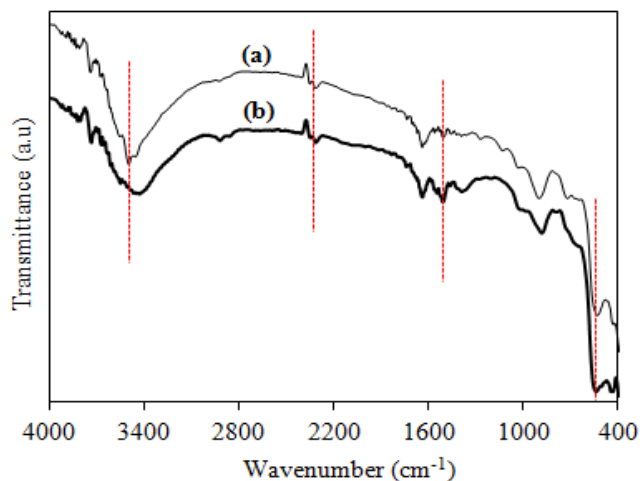


Fig. 2. The FTIR spectra of (a) synthesized ZnO NPs, and (b) commercial ZnO. The commercial ZnO is included for comparison only.

Fig. 2 shows the FTIR spectra of both the synthesized ZnO NPs and commercial ZnO. The FTIR of a commercial ZnO was also measured for comparison. As can be seen, both the synthesized ZnO NPs and the commercial ZnO exhibit very similar spectrum. A characteristic band with strong absorption band at lower energy region ca. 430 cm^{-1} is attributed to the stretching vibration of Zn-O ($\nu\text{Zn-O}$) [23, 24]. Two characteristic bands at ca. 2300 cm^{-1} [23] and 1500 cm^{-1} [20] can be ascribed as stretching vibration mode of C=O ($\nu\text{C=O}$). The peak at ca. 3400 cm^{-1} is assigned to the O-H ($\nu\text{O-H}$) bending vibration of physisorbed water [23]. The FTIR data of the synthesized ZnO NPs indicate its preparation has been successfully achieved in this study. Typical cluster morphology of bright-field-TEM and EDAX images of the synthesized ZnO NPs are shown in **Fig. 3**. In the bright field image, it seems to consist of many round shaped particle with moderated aggregation (**Fig. 3a**) with the average particle size in the range of 7-12.5 nm. The lattice fringes of the ZnO particle is shown in inset of **Fig. 3a**, indicating that the particles of the synthesized ZnO are in high crystal quality [24]. **Fig. 3b** shows a representative EDAX of the synthesized ZnO NPs, the pattern demonstrates the presence of Zn and O. No evidence of other metals impurities can be found except a little amount of Cu from the sample copper grid. The carbonaceous impurity from the preparation step is also observed. In general, the results from the TEM study are in accordance with the XRD data.

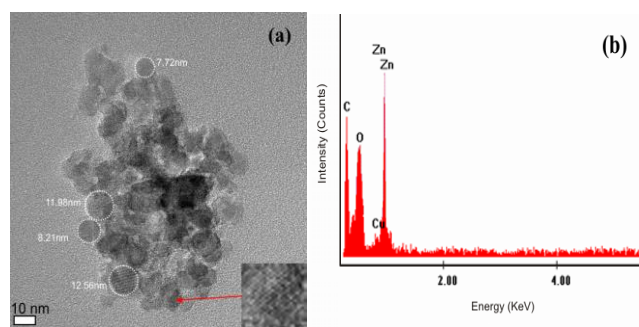


Fig. 3. (a) A typical morphology of a bright-field TEM image (inset: enlarged lattice fringes), and (b) the EDAX pattern of the synthesized ZnO NPs.

Evaluation of MIC of the synthesized ZnO NPs

In this study, activity testing of the synthesized ZnO NPs was conducted to evaluate the effect of reaction parameters including effect of initial concentration of CR dye, dosage of the synthesized ZnO NPs, and contact time on the MIC process for the CR dye in aqueous solution.

Effect of initial concentration of CR dye

The effect of initial concentration of CR dye on the efficiency of the MIC process by the synthesized ZnO NPs was investigated by varying the initial concentration of the CR dye in the range of 10-80 mg/L and the result is shown in **Fig. 4**. As it can be seen, the decolorization efficiency of CR dye was found to decrease with increase the initial concentration of the CR dye. The efficiency decreases from 89.87% to 28.10% as the initial CR dye concentration increase from 10 to 80 mg/L. This result may indicate that the low concentration of CR dye, the low ratio of adsorbate molecule available to be adsorbed in comparison to the number of active site on the surface of the synthesized ZnO NPs. In a word, a high ratio of active site on the surface of the synthesized ZnO NPS is required for high concentration of CR dye. A similar phenomenon related to the effect of catalyst dosage for MIC process has been reported by Xiaoyi *et al.*, [25].

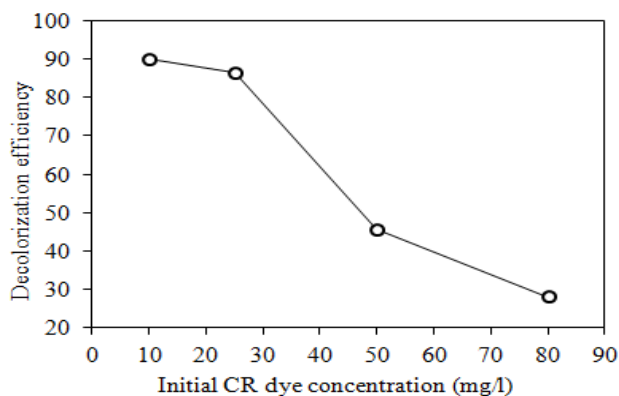


Fig. 4. Effect of initial CR concentration on the catalytic degradation performance of the synthesized ZnO NPs (Conditions: ZnO dosage = 3 g/L, microwave power = 300 watt, and contact time = 3 min).

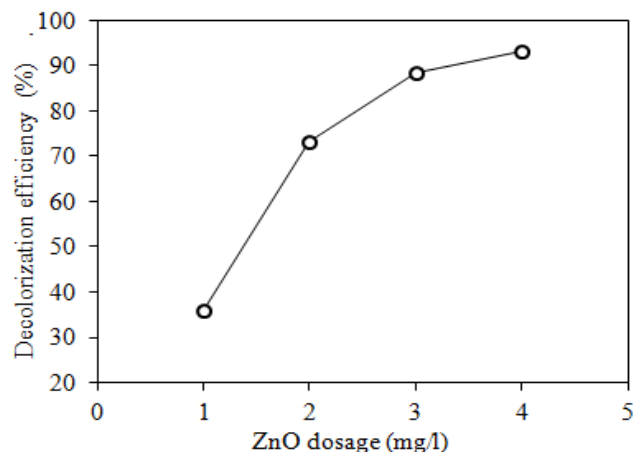


Fig. 5. Effect of dosage on the catalytic degradation performance of the ZnO sample (Conditions: CR dye concentration 10 mg/L, microwave power 300 watt, and contact time 3 min).

Effect of ZnO dosage

The dosage of catalyst is another very crucial factor for catalytic activity because fixed amount of adsorbate can only be absorbed by a given mass of catalyst. The dosage effect was conducted by contacting the dosage of the synthesized ZnO NPs from 0 to 4 g/l to CR dye in aqueous solution and the result is depicted in **Fig. 5**. It can be clearly observed that the decolorization efficiency of the CR dye increases along with the increase the CR dye dosage from 1 to 4 g/l. The higher dosage of the synthesized ZnO NPs, the higher number of active sites on the surface which may lead to more available of CR dye molecules can attach to the ZnO NPs surfaces, advantaging for accelerating the catalytic reaction.

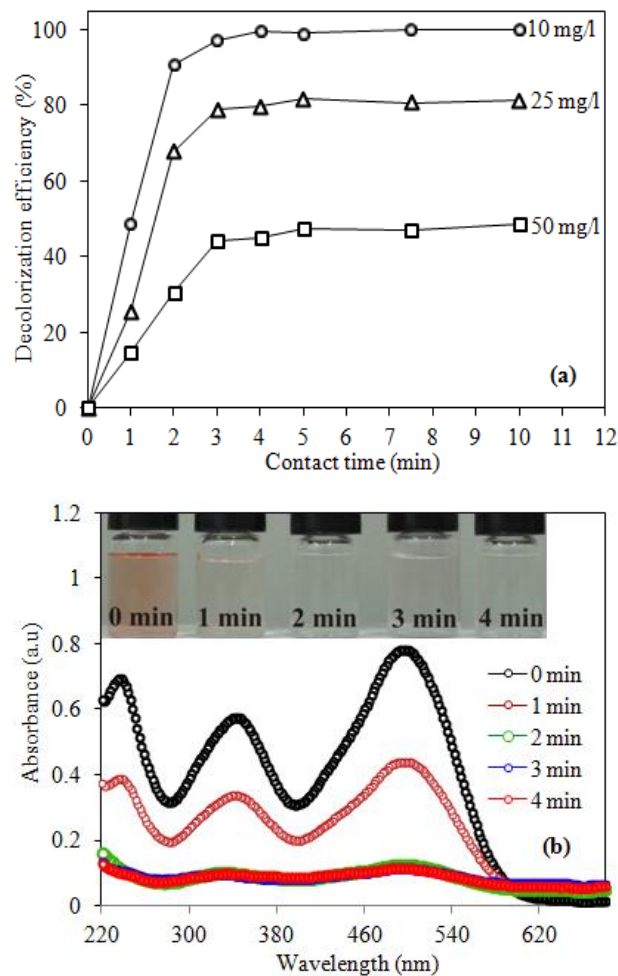


Fig. 6. (a) Effect of contact time on the catalytic decolorization of the CR dye solution in the presence of the synthesized ZnO NPs, and (b) UV-visible spectra changes of 10 mg/L CR dye solution decolorized during MIC process over synthesized ZnO NPs (inset CR dye solution color changes after MIC process).

Effect of contact time

To investigate the effect of contact time on the decolorization efficiency of CR dye, the experiments were carried out with the contact time from 0 to 10 min at different initial concentration of CR dye (10, 25, and 50 mg/l) and the results are presented in **Fig. 6a**. As can be seen, for 10 mg/l of CR dye concentration, 90.63% decolorization efficiency can be fastly achieved within the

first three minute and then the efficiency remains constant with increasing the contact time. A similar trend was observed for 25 and 50 mg/l of CR dye concentration, but the decolorization efficiency for both was lower than that of 10 mg/l, giving the following order: 10 mg/l > 25 mg/l > 50 mg/l. The reason is that the high CR dye concentration requires a high dosage of the synthesized ZnO NPs. **Fig. 6b** shows time-dependent absorption spectra of the CR dye solution during the MIC process in the presence of the synthesized ZnO NPs. It can be seen that the intensity of absorption peak decreases with increase in the contact time, which indicated that the CR dye was decolorized as time prolonged. Inset of **Fig. 6b** shows the color changes of the CR dye solution from appearance of reddish yellow (before MIC process) to a clear solution (after the MIC process) when the synthesized ZnO NPs were used.

The comparison of the decolorization under different treatment process

To observe the microwave effect on the decolorization of CR dye, the testing was conducted under different treatment processes either microwave with (denoted as M-Z) or without (denoted as M-only) the synthesized ZnO NPs. Besides it, both traditional process using water bath agitation procedure (denoted as Trad.) and microwave with commercial ZnO (denoted as M-Z-Com) were also conducted. All the results were compared. As shown in **Fig. 7**, it is obviously observed that the removal efficiency of CR dye under traditional process was lower than the M-Z process, indicating the CR dye removal was more efficient under microwave irradiation. In a word, the microwave can induce the decolorization efficiency of CR dye in aqueous solution. A similar finding has been reported by some previous authors [8-10]. With respect to the CR dye removal efficiency, catalytic activity of ZnO NPs under assistance of microwave irradiation found in this study is obvious in comparison with other reported methods such as catalytic wet oxidation [26], photocatalysis [27], sonocatalysis [28], and biological method [29]. Kondru, *et al.*, [26] reported that the percent color removal of CR dye can be achieved in 45 min using 1 g/l modified Y zeolite as catalyst under optimum pH 7, 90°C using 0.6 ml H₂O₂/350 ml solution. Erdemoğlu, *et al.*, [27] investigated the photocatalytic degradation of CR dye over TiO₂. They found that complete decolorization of CR dye was achieved within 30 min of photo-irradiation. As reported by Wang, *et al.*, [28], the degradation of CR dye using nanometer rutile TiO₂ can be achieved within 180 min under an optimal experimental condition. In addition, biological method for remediation of CR dye at neutral pH using various fungal species has been reported by Bhattacharya, *et al.* [29]. Their results demonstrated that 24 h of incubation time was required to degrade of about 50% of the CR dye. Nevertheless, those reported findings obviously indicated that all the methods used require long reaction time. On the other hand, the MIC method used in this study requires shorter time for removal of the CR dye, implying that the microwave technology give an advantage effect. In term of degradation mechanism, the rapid decolorization of CR dye using ZnO NPs under microwave irradiation may involve non-thermal and thermal effects. Non thermal effect of

microwave irradiation occurs by which the microwave may induce dipole orientation and migration of polar molecule of substances [30], which lead the molecules to higher-excited state and further increase the collision numbers between reactants in the reaction mixture [7]. Thermal effect of the microwave irradiation may follow the mechanism as proposed by Zhang, *et al.* [8] through the formation of “hot spot”. When the ZnO NPs is irradiated with the microwave, at the same time, the surface of ZnO NPs absorbs the microwave energy lead to the formation of “hot spot” having temperature more than 1200°C. Both non-thermal and thermal effects could destroy not only the azo bond but also rapidly degrade the naphthalene ring of the CR dye.

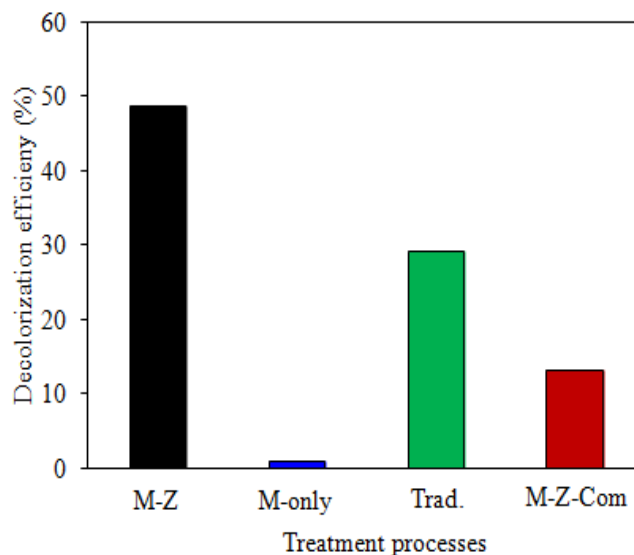


Fig. 7. Effect of different treatment processes on the catalytic decolorization of CR dye (Conditions: CR dye concentration 10 mg/L and contact time 1 min).

In addition, the CR dye treatment under microwave without the synthesized ZnO NPs (M-only) had no a notable effect on the removal of CR dyes. On the other hand, decolorization efficiency of CR dye under microwave with commercial ZnO (M-Z-C) had lower removal efficiency in comparison to the process under microwave with the synthesized ZnO NPs (M-Z), but higher than that of efficiency in the traditional process. The decolorization efficiency of the treatment processes follows the order of M-Z > Trad. > M-Z-C > M-only.

Conclusion

The synthesized ZnO nanoparticle as catalyst has been well characterized using XRD, FTIR and TEM. The experimental results show that the microwave irradiation has significantly improved the efficiency of the synthesized ZnO NPs for decolorization of the CR dye in aqueous solution. Also, results from comparative experiments show that M-Z process exhibit the highest efficiently compared to those for the Trad., M-Z-C, and M-only processes. Overall, the application of ZnO NPs as catalyst in MIC process for treating the synthetic organic dye in industrial effluent will be a promising method for controlling the environmental pollution.

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