Low-temperature synthesis and investigations on photocatalytic activity of nanoparticles BiFeO$_3$ for methylene blue and methylene orange degradation and some toxic organic compounds

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Abstract
The photocatalytic BiFeO$_3$ perovskite nanoparticles were fabricated by gel combustion method using polyvinyl alcohol and corresponding metal nitrate precursors under the optimum mild conditions such as pH 2, gel formation temperature of 80 $^\circ$C, metal/polyvinyl alcohol molar ratio of 1/3, metal molar ratio Bi/Fe of 1/1 and calcination temperature at 500 $^\circ$C for 2 h. The prepared sample was characterized by x-ray diffraction, field scanning electron microscopy, transmission electron microscopy, Brunauer–Emmett–Teller nitrogen adsorption method at 77 K, energy dispersive x-ray spectroscopy, ultraviolet-visible light spectrophotometry, and thermal analysis. The effects of molar ratios of starting material and calcination temperature on phase formation and morphology were investigated. The degradation of methylene blue, methylene orange and some toxic organic compounds such as phenol and diazinon under visible light irradiation by photocatalytic BiFeO$_3$ nanoparticles were evaluated at different parameters and conditions such as the light intensity determined from the light source to the measured sample, the addition H$_2$O$_2$, reaction time and the regeneration performance. Obtained results showed that the synthesized perovskite BiFeO$_3$ nanoparticles for the optimized sample have a size smaller than 50 nm and the high mean surface area of 50 m$^2$ g$^{-1}$. Degradation efficiency was almost 90.0% for methylene blue and 80.0% for methylene orange with added H$_2$O$_2$ after 30 min of reaction. After the 3rd time of regeneration, the BiFeO$_3$ nanoparticles still have 92.8% of the degradation performance for removing methylene blue. Phenol and diazinon toxic compound were degraded with the performance of 92.42% and 85.7%, respectively, for 150 min

Keywords: BiFeO$_3$ nanoparticle, combustion method, toxic organic compound degradation, methylene blue, methylene orange

Classification numbers: 4.02, 5.07
1. Introduction

Previously, in the treatment of water for improving its quality one often applied different chemical, physical, biological methods or their combinations, but their efficiencies were low because of the high cost and the low regeneration performance. Recently, there arose a strong interest in the use of photocatalytic TiO$_2$ which is cost-effective and less hazardous [1, 2]. However, TiO$_2$ has some disadvantages such as high bandgap (approximately 3.2 eV) equivalent to the absorption wavelength in the ultraviolet range, and being difficult to recover and to regenerate [3, 4]. Although there have been various studies on TiO$_2$ for absorbing visible light, there has been little research into the photocatalytic activities of doped TiO$_2$. Recently, in references [5–7] bismuth ferrite BiFeO$_3$ with the bandgap around 2.1 eV [8] was prepared and investigated. It is an easily reusable material and has magnetic properties at room temperature [9, 10]. The previous research was focused on solid state reaction at high temperature [11], and the single phase of perovskit BiFeO$_3$ nanoparticles was difficult to prepare due to the volatility of Bi$_2$O$_3$. Therefore, in recent studies the authors followed the strategy to synthesize single phase perovskite BiFeO$_3$ with low temperature [12–14] and to study the regeneration of the photocatalytic activity on organic degradation [5, 15–17].

In the present work we synthesize single phase perovskite BiFeO$_3$ nanoparticles via polymeric precursors prepared by using polyvinyl alcohol (PVA) at relatively low temperature 500 °C. Photocatalytic activity and regeneration of BiFeO$_3$ nanoparticles on the degradation of methyl blue, methyl orange, phenol and diazinon with various conditions also were studied.

2. Experimental

2.1. Chemicals

All agents were analytical grade and as received without further purification. Fe(NO$_3$)$_3$·6H$_2$O, Bi(NO$_3$)$_3$·9H$_2$O, HNO$_3$, KOH, PVA, methylene blue, methylene orange, phenol, diazinon, a solution of H$_2$O$_2$ 5% were purchased from Sigma-Aldrich and Merck.

2.2. Synthesis of BiFeO$_3$

PVA used in gel combustion synthesis of BiFeO$_3$ is water soluble and has hydroxyl ligands as side group which provides complexing sites to metal ions [18, 19]. Fe(NO$_3$)$_3$ and Bi(NO$_3$)$_3$ were mixed together in different proportions in a molar to give a concentrated solution. PVA solution was obtained by dissolution in water at 80 °C then the metal solution was added to the PVA solution to have the suitable amount. The ratio of metal and PVA was applied 1/3 under the pH of 2. The solution was continuously stirred with a magnetic stirrer to remove the excess of water and turned into a very viscous and clear transparent brown-red-colour gel. A homogeneous solution was proved by the clearness of the solution. The viscous gel was dried out for 4 h in an air oven at 120 °C, then the product with no turbidity or precipitation was calcinated at the suitable temperature in air for 2 h to obtain the perovskite-like single phase BiFeO$_3$.

2.3. Characterization methods

The products obtained during different stages were characterized by x-ray diffraction (XRD) using Siemens D-5000 diffractometer (Germany) with Cu-K$_\alpha$ radiation ($\lambda = 0.154059$ Å) in the range of 2θ = 10°–95°, and a scanning rate of 0.02° s$^{-1}$. The average crystalline size of the BiFeO$_3$ was calculated from the half-width of the ceria (111) peak according to the Scherrér’s equation, where the Scherrér constant was taken as 0.89. The micromorphology of the nanoparticle was evaluated by field emission scanning electron microscopy (FE-SEM) of Hitachi S-4800 microscope (Japan) and transmission electron microscopy (TEM) of JEOL JEM-1010 (USA). The surface chemical composition (EDS) of the sample was determined by Hitachi S 4800 spectrometer. Thermogravimetric analysis and differential thermal analysis (TGA-DTA) diagrams of the gel precursors were carried out on a Setaram Labsys EVO (France) from room temperature to 900 °C in the air with a heating rate of 10° min$^{-1}$. The specific area was determined by using nitrogen adsorption at 77 K and the linear part of the Brunauer-Emmett-Teller (BET) model, the average pore size was calculated by using the Barrett-Joyner-Halenda (BJH) formula, and the Quantachrome Autosorb-iQ Station 1 (USA).

2.4. Photocatalytic activity investigation

Methylene blue (MB), methylene orange (MO) and some toxic organic compounds were prepared in various concentrations, and the solutions were settled down in dark. Then the prepared solutions were irradiated together with the photocatalytic material under different conditions in the visible light by using Ace photochimical power supplies and mercury vapor lamps (USA) with 450 W (7825-3) lamp in a 50 mm quartz well. The tested solution was maintained constant throughout by a cooling circulating system. The concentrations of MB, MO and toxic organic compounds before and after the reaction were determined by using the photometric colorimetric method and the UV-1800 Shimadzu spectrophotometer (Japan). After the reaction, the catalyst was separated by centrifugation. The absorbance A$_0$ measured after stirring for 1 h in the dark was taken as the quantity proportional to the initial concentration C$_0$, and the absorbance A$_t$ measured after variable periods was taken as the quantity proportional to the residual concentration C$_t$. The degradation efficiency of the material was calculated by the formula

$$H(\%) = \frac{C_0 - C_t}{C_t} \times 100 = \frac{A_0 - A_t}{A_t} \times 100.$$
3. Results and discussion

3.1. BiFeO₃ synthesis

In this PVA gel combustion synthesis of BiFeO₃, some process conditions such as calcination temperature and Bi/Fe molar ratio of the phase of perovskite BiFeO₃ were investigated. The other conditions like pH, gel formation temperature were indicated in the previous works [12–14].

Figure 1 shows XRD diagrams of the synthesized samples with metal/PVA molar ratio of 1/3 calcined at 250 °C, 450 °C, 500 °C and 550 °C for 2 h. For the case of the samples calcined at 250 °C, no crystalline phase was observed which corresponded to the amorphous powder. The typical peak represented for the crystalline BiFeO₃ occurred at a temperature of 450 °C. However, the signal was more clearly shown when increasing temperature was kept to reach 500 °C. XRD diagrams of samples calcined at 500 °C showed no peaks attributable to Bi₂O₃ and Fe₂O₃, and the products are pure perovskite oxide with the orthorhombic single phase of perovskite type BiFeO₃. All the diffraction peaks coincided with those of standard pattern (JCPDS card No. 86-1518). There was a peak attributed to the β-Bi₂O₃ phase in x-ray diffraction diagram at a calcination temperature of 550 °C. This means that the perovskite structure of BiFeO₃ was destroyed in air to form a single phase of metal oxide.

The metal molar ratio of the Bi/Fe has a strong influence on the perovskite formation of BiFeO₃. The XRD patterns of the synthesized material with different Bi/Fe of 5/1, 3/1, 1/1, 1/3, 1/5 and calcination temperature of 500 °C were illustrated in figure 2. It can be clearly seen that if the metal molar ratios of Bi/Fe differ from 1/1, then there exist different phases such as β-Bi₂O₃, Bi₃Fe₂O₇, and α-Bi₂O₃ [20]. At the Bi/Fe ratio of 1/1, only orthorhombic single phase of BiFeO₃ perovskite type was observed.

3.2. Characterization of synthesized BiFeO₃

According to the representative results for the factors influenced on phase perovskite formation of BiFeO₃, the optimum process conditions are calcination temperature of 500 °C in air, metal molar ratio Bi/Fe of 1/1, gel formation of 80 °C, metal/PVA molar ratio of 1/3, and solution pH 2.

3.2.1. TG–DTA analysis. The TG and DTA diagrams of the gel precursor illustrated in figure 3 which have two discrete weight losses were obtained around 122.11 °C and 301.9 °C. It has been proved by two endothermic peaks in the DTA curve. The first loss of weight (7.64%) was in a range of 70 °C to 130 °C accompanied by a peak near 122.11 °C in DTA curve caused by the loss of surface absorbed water or residual water in the increasing temperature processes. The major weight loss (25.49%) between 280 °C and 450 °C with the maximum at 301.9 °C was due to the oxidation decomposition of the PVA and the decomposition of the nitrate of the precursor. Besides, the DTA curve has shown an exothermic peak near 325.56 °C that might be the perovskite type formation of BiFeO₃ from the amorphous component. At the temperature of 400 °C, there was no changing weight of the samples, which corresponded to the stabilized perovskite type of BiFeO₃. That means the TG-DTA curves have a strong agreement with the XRD spectra in figure 1.

The BiFeO₃ material under the optimum process conditions is in the orthorhombic single phase of the...
perovskite type, as reported in some papers using the solvothermal method [16, 21]. So, the single phase of the perovskite type BiFeO$_3$ was synthesized by gel combustion method successfully under a low temperature of 500 °C with the easily prepared initial components. This temperature is lower than the temperature used by Hengky et al [11] and the prepared nanomaterials have smaller size than the one reported by Gao et al [5].

3.2.2. BET surface area, SEM, TEM images, XRD and EDS. BET surface area of calcined powder was found to be 50 m$^2$ g$^{-1}$. The data obtains of the nanoparticle BiFeO$_3$ that could be applied in catalysis and adsorption.

TEM and SEM micrograph images of the BiFeO$_3$ particles provide the information about their size and morphology. It can be seen clearly from the figure 4 that the homogeneous morphology of the sample was again proved by x-ray diffraction (figure 5) and the nanosize of the particle in a range smaller than 50 nm was observed. Figure 6 illustrates the TEM micrograph image of the BiFeO$_3$ nanoparticles in 100 nm scale.

EDS has again confirmed the composition of the products. Figure 7 shows the EDS spectra of BiFeO$_3$ particles. The EDS spectrum pointed out the presence of bismuth, iron, and oxygen of the prepared sample. The

**Figure 4.** FE-SEM images of the BiFeO$_3$ powder sample with Bi/Fe molar ratio of 1/1 calcined at 500 °C for 2 h.

**Figure 5.** X-ray diffraction of the BiFeO$_3$ powder sample with Bi/Fe molar ratio of 1/1 calcined at 500 °C for 2 h.

**Figure 6.** TEM images of the BiFeO$_3$ powder sample with Bi/Fe molar ratio of 1/1 calcined at 500 °C for 2 h.

**Figure 7.** EDS analysis of the BiFeO$_3$ powder sample with Bi/Fe molar ratio of 1/1 calcined at 500 °C for 2 h.
composition of the elements in the sample obtained by the PVA-gel combustion method was 67.21%; 16.36%; and 15.74% for bismuth; iron and oxygen, respectively, in the agreement with theoretical calculation (table 1).

### 3.3. Visible light photocatalytic activity of MB and MO

The nanoparticle perovskite type BiFeO$_3$ after synthesis under optimum conditions was used to investigate the photocatalytic activity for the MB and MO degradation. The nanomaterial was studied at different conditions under the simulated natural light systems.

#### 3.3.1. Photocatalytic performance in the darkness

Both solutions MB and MO were unchanged after 24 h. So, the perovskite nanoparticles BiFeO$_3$ in the darkness for 24 h has not been able to degrade MO and MB solution.

#### 3.3.2. Photocatalytic activity under the visible irradiation

With the ratio of materials/solution of 1.0 g L$^{-1}$ and the initial concentration $C_0$ = 10 ppm of MB and $C_0$ = 10 ppm of MO, the prepared sample was stirred 30 min during the experiment. The investigation of the photocatalytic activity of the nanoparticles was described in figure 8. The figure shows that the degradation performance of the material for MB over the first 15 min was only 26.0%, then it increased to reach 90.0% when the reaction time was 3 h. Whereas the MO degradation is very low around 6.0% after 15 min of the illumination and it turns out to reach a peak at 20.0% (table 2). So, to increase the photocatalytic activity we added H$_2$O$_2$ to the reaction solution. The photodegradation of the MO and MB by the perovskite BiFeO$_3$ was assemble as follow [22] and figure 9:

\[
\begin{align*}
BiFeO_3 + h\nu &\rightarrow BiFeO_3(e^- (CB)) + H^+(VB), \\
h^+ + H_2O (ads) &\rightarrow OH^+ (ads) + H^+,
\end{align*}
\]

\[
\begin{align*}
o_2 + e^- (CB) &\rightarrow O_2 (ads), \\
o_2^+ (ads) + H^+ &\rightarrow HOO^+(ads), \\
2HOO^+ (ads) &\rightarrow H_2O_2 (ads) + O_2, \\
H_2O_2 (ads) &\rightarrow 2OH^- (ads), \\
dye + OH^- &\rightarrow CO_2 + H_2O.
\end{align*}
\]

#### 3.3.3. Additive H$_2$O$_2$ effect

A small amount of 0.1 ppm of H$_2$O$_2$ was added to a series of solutions prepared in different concentrations from 5 ppm to 30 ppm of MB and from 5 ppm to 10 ppm of MO. The photocatalytic activity of BiFeO$_3$ was illustrated in table 3. There was a difference in degradation efficienciesy of MB and MO. For the MB with the concentrations from 5 ppm to 15 ppm, the degradation efficiency reached 99.0% after 15 min of illumination. In the MB solutions of 20 ppm and 30 ppm concentrations, the remained concentration of MB after 30 min and 40 min of illumination was changed to 15.38% and 20.0%, respectively. Moreover, the MB solutions of 5 ppm and 10 ppm were changed to 63.50% and 84.30% after 15 min of the illumination and it turns out to reach a peak at 90.0% (table 2). So, to increase the photocatalytic activity we added H$_2$O$_2$ to the reaction solution. The photodegradation of the MO and MB by the perovskite BiFeO$_3$ was assemble as follow [22] and figure 9:

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H_2O_2 (ads) &\rightarrow 2OH^- (ads), \\
dye + OH^- &\rightarrow CO_2 + H_2O.
\end{align*}
\]

### Table 1. The chemical composition of the BiFeO$_3$

| Elements  | Bi   | Fe   | O   |
|-----------|------|------|-----|
| Theory percentage (%) | 66.77 | 17.90 | 15.33 |
| Analytical percentage (%) | 67.21 | 16.36 | 15.74 |

### Table 2. Photocatalytic performance of the methylene blue and methylene orange degradation.

| Time (min) | Degradation (%) of MB | Degradation (%) of MO |
|-----------|------------------------|------------------------|
| 15        | 26.0                   | —                      |
| 30        | 29.42                  | 9.62                   |
| 45        | 33.96                  | —                      |
| 60        | 51.57                  | 12.30                  |
| 75        | 56.90                  | —                      |
| 90        | 63.50                  | 15.38                  |
| 120       | 80.0                   | 20.0                   |
| 150       | 84.30                  | —                      |
| 180       | 90.0                   | —                      |

Figure 8. Adsorbed performance of BiFeO$_3$ powder sample with Bi/Fe molar ratio of 1/1 calcined at 500 °C for 2 h under visible light: (a) the MB and b) the MO solution.
illumination, respectively, was less than 1%. Whereas for the MO solution with concentration of 5 ppm, the degradation efficiency reached 97% after 45 min of illumination. In the case of MO with higher concentration (10 ppm), the degradation efficiency about 64.5% after 120 min under the same condition.

3.3.4. Light irradiation distance effect. The other factor that has a strong influence on the photocatalytic activity of the nanomaterial perovskite BiFeO₃ is the distance of the illumination. Table 4 revealed the reflectance of the light distance on the MB and MO degradation performance. Despite over 99.0% of MB was decomposed after 45 min with no distance from the solution to the light source, there was only 80.0% of efficiency with the 15 cm of lighting distance after 2 h. The MO decomposition was also reported with higher efficiency around 53.0%, and 66.2% after 60 min and 90 min respectively of the treatment if the distance was zero. However, only 20.0% of MO was degraded correspond to 15 cm light-distance reaction.

3.4. Regenerative performance

The recovered nanoparticles were dehydrated for 2 h in the air with the temperature of 120 °C, then the product was used again to degrade the MB solution with concentration of 20 ppm. A small amount of H₂O₂ was added to the solution for the first time of regeneration and the illumination took place for 30 min. The degragation performance after the use of nanomaterial for the first times decreased by only 0.1%. After the second and third times of reusing it decreased to to 99.0% and 92.80%, respectively. These results clearly demonstrated the regeneration ability of BiFeO₃ nanoparticles.

The UV-visible curve in figure 10 shows the adsorption of the nanoparticle BiFeO₃ in the range visible irradiation after third-time regeneration. The efficiency of degradation of

![Figure 9. Proposed mechanism of photocatalytic reaction on BiFeO₃.](image)

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**Table 3.** Effect of adding H₂O₂ on methylene blue and methylene orange degradation.

| Solution concentration | Degradation performance (%) during time |
|------------------------|----------------------------------------|
|                        | 15 min | 30 min | 45 min | 60 min | 120 min |
| MB 5 ppm               | >99    | —      | —      | —      | —       |
| MB 10 ppm              | >99    | —      | —      | —      | —       |
| MB 15 ppm              | >99    | —      | —      | —      | —       |
| MB 20 ppm              | 93.80  | >99    | —      | —      | —       |
| MB 30 ppm              | 78.30  | 91.40  | >99    | —      | —       |
| MO 5 ppm               | 63.0   | 81.10  | >97    | —      | —       |
| MO 10 ppm              | 23.80  | 37.80  | 43.70  | 51.20  | 64.50   |

**Table 4.** Distance effectiveness on the methylene blue and methylene orange degradation.

| Solution concentration | Light distance d (cm) | MB and MO degradation efficiency (%) during time |
|------------------------|-----------------------|-----------------------------------------------|
|                        | 15 min | 30 min | 45 min | 60 min | 90 min | 120 min |
| MB 10 ppm              | 15     | 26.0   | 29.42  | 33.96  | 51.57  | 63.50   | 80.0    |
|                        | 0      | 47.20  | 74.80  | >99    | —      | —       | —       |
| MO 10 ppm              | 15     | —      | 9.62   | —      | 12.30  | 15.38   | 20.0    |
|                        | 0      | 26.10  | 36.7   | 43.5   | 53.0   | 66.20   | —       |
the BiFeO$_3$ powder was slightly decreasing after triple times of using for one sample from over 99.9% to 92.8%.

3.5. Toxic organic compounds degradation

To study the further application of the material, two toxic organic compounds were chosen to be decomposed under the visible light: phenol at concentration of 500 ppm and diazinon at concentration of 1 ppm. We assumed that the organic matter was degraded completely to CO$_2$ and H$_2$O form as $\text{(electrochemical reaction)}$. The total organic chemical was calculated by Walkley–Black methods. The decomposition performance of the phenol and diazinon in solution by the perovskite BiFeO$_3$ single phase nanocrystalline powder was showed in figure 11.

From the results in figure 11, it can be seen clearly that when the time increases from 15 min to 150 min, the degradation performance of phenol and diazinon increases from 25.5% to 92.42% and from 15% to 85.7%, respectively. It also can be concluded that the reaction of both organic compounds happened faster in first 60 min, 62.6% of phenol and 43.3% of diazinon were degraded. The photodecomposition process of phenol occurred with higher efficiency. Approximately 93% of phenol was degraded after 150 min of illumination which is much higher than the CuO/CeO$_2$ performance by Massa et al [23], whereas 85.7% of diazinon, which similar to N-doped TiO$_2$ system by Asadi et al [24] was decomposed at the same condition.

4. Conclusions

The single phase nanocrystalline powder of the BiFeO$_3$ with the average nanoparticle size $<50$ nm, a surface area of $50 \text{m}^2 \text{g}^{-1}$ was successfully synthesized from polymeric precursors made by polyvinyl alcohol as homogenizer under low temperature of $500^\circ\text{C}$ and optimum conditions: pH 2, gel formation temperature of $80^\circ\text{C}$, metal molar ratio Bi/Fe of 1/1 and metal/PVA of 1/3. The photocatalytic activity and the regeneration performance of prepared nanomaterial was also studied for methylene blue and methylene orange degradation under the visible light irradiation. The nanoparticle perovskite BiFeO$_3$ showed a high photocatalytic activity to decompose the organic pollutants: over 90.0% of MB was removed under the visible irradiation for 3 h and 99.0% with additive H$_2$O$_2$ for 30 min while around 66.0% of MO was cleared from wastewater for 2 h. After the 3rd time of regeneration, the material also able be used by the evidence of 92.8% of removing MB with adding H$_2$O$_2$. During 150 min of the reaction, 92.42% of phenol and 85.7% of diazinon were decomposed.

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References

[1] Paola A D, García-López E, Marcì G and Palmisano L 2012 J. Hazard. Mater. 211 3
[2] Ullah I, Ali S, Hanif M A and Ali Shahlir S 2012 Int. J. Chem. Biochem. Sci. 2 60
[3] Hernández-Alonso M D, Fresno F, Suárez S and Coronado J M 2009 Energy Environ. Sci. 2 1231
[4] Factorovich M, Guz L and Candal R 2012 Adv. Phys. Chem. 2011 1
[5] Gao F, Chen X, Yin K, Dong S, Ren Z, Yuan F, Yu T, Zou Z and Liu J M 2007 Adv. Mater. 19 2889
[6] Timmaji H K 2012 Bismuth-based oxide semiconductors: mild and practical applications PhD Dissertation The Univeristy of Texas at Arlington
[7] Wei J, Li H, Zhang C, Xu Z, Mao S and Dkhil B 2012 J. Mater. Sci., Mater. Electron. 23 1869
[8] Kubel F and Schmid H 1990 Acta Crystallogr. B 46 698
[9] Seidel J et al 2009 Nat. Mater. 8 229
[10] Yang Y C, Liu Y, Wei J H, Pan C X, Xiong R and Shi J 2014 RSC Adv. 4 31941
[11] Hengky C, Dunn S, Singh V and Loh L 2010 NSTI-Nanotech. 1 570
[12] Jiang H, Yasutaka M, Nobuhiko K, Yoshinori Y, Takahiro T and
Nobukazu K 2008 J. Ceram. Soc. Japan 116 837
[13] Jiang H, Dai H, Meng X, Zhang L, Deng J, Liu Y and Au C T
2012 J. Environ. Sci. 24 449
[14] Kim J K, Kim S S and Kim W J 2005 Mater. Let. 59 4006
[15] Luo W, Zhu L, Wang N, Tang H, Cao M and She Y 2010
Environ. Sci. Technol. 44 1786
[16] Li S, Lin Y H, Zhang B P, Nan C W and Wang Y 2009 J. Appl.
Phys. 105 56105
[17] Nguyen B H and Nguyen V H 2015 Adv. Nat. Sci.: Nanosci.
Nanotechnol. 6 043001
[18] Saha S K, Pathak A and Pramanik P J 1995 Mater. Sci. Lett.
14 35
[19] Pradhan S K and Roul B K 2012 J. Sol-Gel Sci. Technol.
64 375
[20] Wang P W, Guttag M and Tu C S 2014 J. Surf. Eng. Mater
Adv. Technol. 4 295
[21] Yuning H, Jin Y and Zhang Y 2010 J. Molecular Catal. A:
Chem. 331 15
[22] Ajmal A, Majed I, Malik R N, Idriess H and Nadeem M A
2014 Rsc. Adv. 4 37003
[23] Massa P, Ivorra F, Haure P and Fenoglio R 2011 J. Hazard.
Mater. 190 1068
[24] Salarian A A, Hami Z, Mirzaie N, Mohseni S M, Asadi A,
Bahrami H, Vosoughi M, Alinejad A and Zare M R 2016
J. Mol. Liq. 220 183