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Enhanced photodegradation of hazardous tartrazine by composite of nanomolecularly imprinted polymer-nanophotocatalyst with high efficiency

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ABSTRACT

In this research, a nanocomposite of molecularly imprinted polymer (MIP)-nanophotocatalyst for enhanced photodegradation of tartrazine with high efficiency and nanocomposite of non-imprinted polymer (NIP)-nanophotocatalyst were synthesized by precipitation polymerization and it was carried out in an aqueous medium to employ a green method. Acrylamide as functional monomer, N,N´-methylene-bis-acrylamide as cross-linking agent, tartrazine as a template, and TiO_2 as a nanophotocatalyst were used. The synthesized nanocomposites (MIP-TiO_2 and NIP-TiO_2) were characterized by using thermal gravimetric analysis, scanning electron microscopy, transmission electron microscopy, and Fourier transform infrared spectroscopy. The MIP nanocomposite as an effective sorbent was used to extract and photodegrade tartrazine from water media. The effects of pH, time, and concentration of tartrazine solution and kinetics of photodegradation of tartrazine were investigated. The best pH and the optimum time for tartrazine photodegradation in a 10 ml solution containing tartrazine (3×10^{-5} M) and nanocomposite (1 mg) were obtained to be 2 and 5 h, respectively. The results showed that the MIP nanocomposite has improved the photodegradation of tartrazine by 100% compared to NIP nanocomposite and pure nanophotocatalyst which yielded at 63 and 56%, respectively, under the same condition.

Keywords: Enhanced photodegradation; MIP nanocomposite; Tartrazine; NIP nanocomposite; Green synthesis; TiO_2 nanophotocatalyst

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1. Introduction

One of the major concerns of today's world is water pollution and contamination. Increase in industrial wastewater from textile, food, drug, etc. containing different types of dyes as such makes it more disconcerting. Strongly colored effluents not only create environmental problems, but can also affect the human life as most of them are carcinogenic and mutagenic. The reports show that it is estimated that 10–15% of the dye is lost in the effluent during the dyeing process. The discharge of such effluents into the ecosystem is a source of visual pollution and disorder for aquatic life [1–3]. Water-soluble dyes are commonly used as coloring agents in a variety of products; even at very low concentrations they are highly visible. These dyes are considered as non-oxidizable by conventional physical and biological treatments because of their complex molecular structure and the large size of their molecules. Thus, their decolorization is one of the necessary processes in wastewater treatment [4]. Nowadays, because of these problems, it is urgently required to develop highly effective and sensitive methods to remove pollutants from water environment. In order to remove pollutants such as azo dyes and ions from wastewater, different kinds of method such as photo catalytic degradation [5,6], hetero catalytic phenton [7], electrochemical removal [8], and different kinds of adsorbent such as adsorbents prepared from industrial waste [9–13], carbon nanotube composite [14,15] and natural adsorbents [16–18], and bottom ash and de-oiled soya [19–26] were used.

Some of these dyes used in the food industry have acceptable daily intake values that are strictly controlled by laws and regulations in various countries [27]. The water soluble acidic azo dye, tartrazine (FD & C Yellow No. 5, EEC Number = E102, CI Number = 19140) is chemically the trisodium 5-hydroxy-1-(4-sulfonatophenyl)-4-(4-sulfonatophenylazo)-H-pyrazol-3-carboxylate. It is a water-soluble powder which is vastly used in food products, drugs, cosmetics, and pharmaceuticals [28,29]. The acceptable daily intake per person is 0–7.5 mg/kg body weight [30]. Tartrazine is found as a food additive in the following foodstuffs: Shells of medicinal capsule, syrups, cosmetics, flavored chips, custard powder, soups, sauces, jam, mustard, yogurt, and many convenience foods, including glycerin, lemon, and honey products, also in hair products, moisturizers, crayons, stamp dyes, vitamins, and antacids [31–35]. Most of the azo dyes appear to cause allergic and/or intolerance reactions. Tartrazine will also catalyze ADHD syndrome [36], asthma [37], migraine and thyroid cancer [38], and other reactions can include blurred vision, itching, purpura, eczema, mutogenetics, and carcinogenetic [39]. Because of its hazardous health effect, food and drink containing tartrazine should be avoided [4]. In spite of restrictions, some countries still use this color and due to its harmful effects it must be removed from wastewater.

In order to remove tartrazine from water, several methods are used such as polystyrene anion exchange resins [2], hen feathers [4], magnetic molecularly imprinted polymer (MIP), and [40] photodegradation [3,41]. As seen basically these methods can basically be divided into two major groups, adsorption and degradation, that each group has their advantages and disadvantages.

The MIP is one of the effective adsorption methods. MIP is a convenient and powerful technique for preparing polymeric materials with artificial receptors. The MIP technique is based on the molecular memory of the substrate to be recognized (template) and memorized in polymeric materials during their preparation. Subsequently, the extraction of the template from the polymer gives specific recognition sites. The polymer containing sites with high affinity for the template has different applications such as removal and extraction of dyes from a water environment [42–45]. However, extraction methods only transfer dyes from the liquid to the solid phase causing secondary pollution and requiring further treatment [46].

Another method is an advanced oxidation process which is one of the most powerful methods for the removal of pollutants from water. Photocatalytic oxidation using a semiconductor such as TiO_2 as photocatalyst is one of the cleanest, greenest, and the most inexpensive technologies for degradation of hazardous organic and inorganic [47] pollutant from wastewater. There are three different types of $TiO_{2,}$ but the adsorptive affinity of the anatase type for degradation is larger than the rutile and the brookite type [3]. The photocatalysts have very poor selectivity and cannot differentiate between the pollutants [48]. Thus, it is noteworthy to improve the selectivity of photocatalyst. Several methods were proposed to improve the selectivity of TiO_2. First is to control the electric charge by adjusting pH, but this method is not useful enough because most pollutants are neutral [49]. The other way is to modify the surface of TiO_2 by arginine to reach an effective adsorption [50], but it is reported that this type of photocatalyst is not stable enough under UV light and the organic host itself could be decomposed [51]. Nowadays, a certain method is employed to imprint the cavities of the target onto the surface of the photocatalyst so the pollutant is adsorbed and degraded on the surface. To reach this goal, previous studies have comprised a method in which the surface of TiO_2 should be modified first and then for MIP synthesis, surface polymerization should be carried out [48,51]. But in this method, a pre-modification of photocatalyst is not simply done.

As mentioned above, pure nanophotocatalysts are not selective sorbents and they have a low capacity. Using the photocatalytic technology and MIP for effective photodegradation in water environment will not only modify the photocatalytic materials, but would also improve the selectivity of the photocatalyst. We have used a simple precipitation polymerization to incorporate the TiO_2 into the matrix of MIP without any pre-modification. In order to increase the efficiency of photodegradation we have synthesized the nanocomposite of MIP-nanophotocatalyst. In our

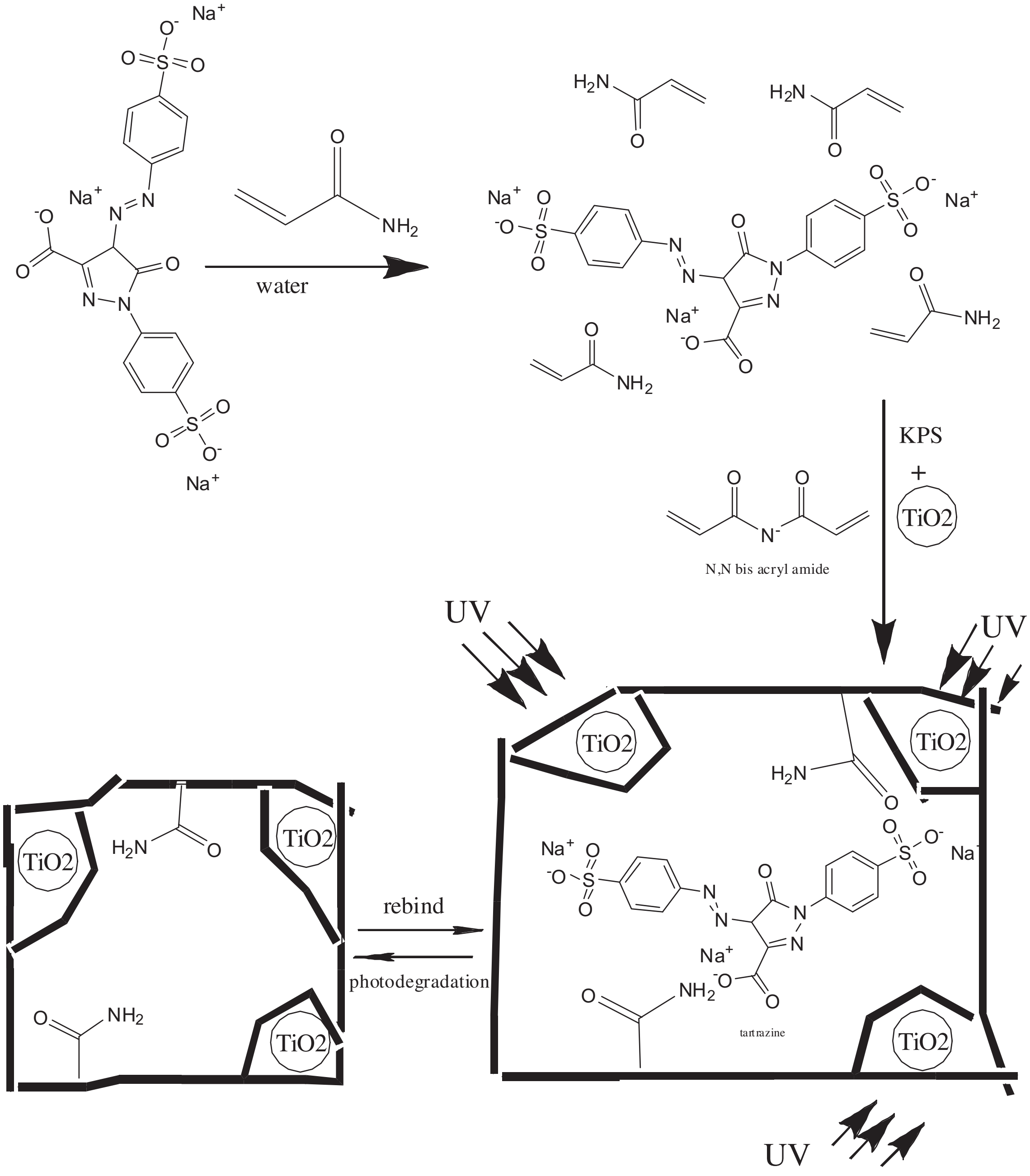

Fig. 1. Schematic synthesis of MIP nanocomposite.

previous work [52], we synthesized and characterized MIP and non-imprinted polymer (NIP) of tartrazine with high efficiency. The present study denotes a new effective nanocomposite for the extraction and photodegradation of tartrazine in water media. For this purpose, new nanocomposite MIP-TiO_2 of tartrazine with high efficiency and nanocomposite of NIP–TiO_2 were synthesized by precipitation polymerization in aqueous medium as a green method and were characterized. It is important to control the particle size of the sorbent for different applications such as removal and separation, precipitation polymerization which causes the uniformly sized polymers. For synthesizing nanocomposites, acrylamide (AA) as a functional monomer, N,N´-methylene-bis-acrylamide as a cross-linking agent, tartrazine as a template, and TiO_2 as a nanophotocatalyst were used. Characterizations of the nanocomposites were carried out by scanning electron microscope (SEM), transmission electron microscope (TEM), thermal gravimetric analysis (TGA), and Fourier transform infrared spectroscopy (FT-IR). The MIP nanocomposite as an effective sorbent was used for extraction and photodegradation of tartrazine from a water environment under UV irradiation. The effects of pH, time, and the concentration of tartrazine solution were optimized to reach the best photodegradation condition.

2. Experimental section

2.1. Chemicals and materials

Tartrazine sodium chloride with a purity of 98% was used as a yellow powder, freely soluble in water, and was purchased from the ministry of health and medical education (Tehran, Iran). AA, N,N´-methylene-bis-acrylamide, potassium persulfate, and other chemicals were of analytical reagent grade and obtained from Merck (Germany). TiO_2 nanoparticles (P25) were obtained from Evonik Industries.

2.2. Synthesis of MIP and NIP nanocomposites

The schematic representation of tartrazine imprinting/degradation from the nanocomposite is shown in Fig. 1. The nanocomposite of tartrazine was prepared by non-covalent approach. The mole ratio of template/functional monomer was 1:2. In a typical preparation, Tartrazine (1 mmol, 0.265 g) as the template and AA (2 mmol, 0.07 g) as the functional monomer were dissolved in 40 ml of distilled water and the solution was stirred for 2 h to pre-organize. N, N´-methylene-bis-acrylamide (10 mmol, 0.77 g), as a cross-linking agent, and TiO_2 (0.4 g) as a photocatalyst and potassium per sulfate (0.185 mmol, 0.025 g) as an

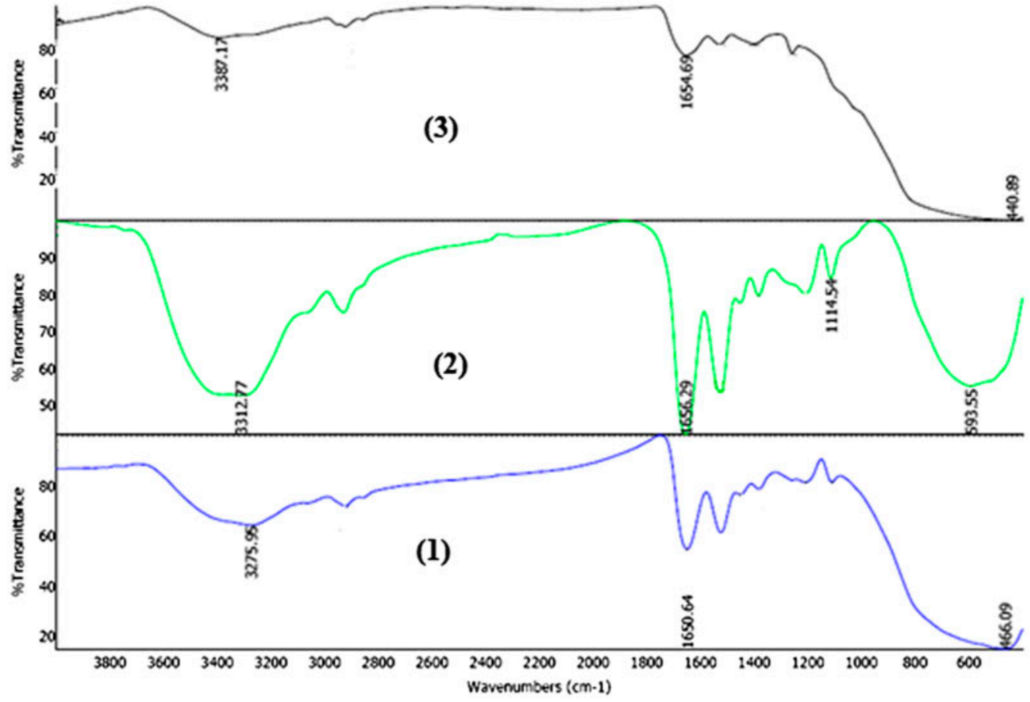

Fig. 2. FT-IR spectra of washed MIP nanocomposite (1), non-washed MIP nanocomposite (2) and washed NIP nanocomposite (3).

initiator were added to the mixture and then was dispersed by sonication. After sonication, the mixture was purged with N_2 (pressure: 2.5 bar) for 10 min and then the Pyrex glass tube (length: 22 cm, internal diameter: 4 cm) was sealed under N_2 atmosphere. Afterwards, the glass tube was stirred in a water bath maintained at 70ºC for 22 h and to avoid the precipitation of TiO_2, the solution was stirred by a magnetic stirrer during the polymerization. The produced nanocomposite was filtered using a centrifuge. The obtained particles were washed with hot distilled water/ammonia solution (8: 2, v/v), and followed by extraction using hot water for three times. The complete removal of tartrazine was investigated by a UV spectrophotometer (PERKIN-ELMER) at 428 nm until tartrazine spectra disappeared. Extractions of templates from the nanocomposite have created selective cavities. The NIP–TiO_2 was also exactly synthesized with the same procedure, but without the template to show the non-specific binding in nanocomposite matrix.

2.3. Characterization

FT-IR spectra of nanocomposite polymers were recorded to study functional groups (Bruker model EQUINOX 55). The TGA of the polymer was carried out on model PL-STA-1500 instruments from Polymer Laboratories' Company (Church Stretton, Shropshire, UK) to study the thermal behavior of composite. SEM and TEM were used to study the morphology of polymer particles (Philips XLC and Philips C30). Wavelength-Dispersive X-ray spectroscopy (WDX) was used to study the map of individual elements (Philips XLC). Total organic carbon analyzer (TOC) was used to display the organic carbon amount after degradation (Rosemount, Dohrmann DC-190).

2.4. Photocatalytic degradation procedure

A measure of tartrazine in aqueous solution was investigated in batch experiments. 1 mg of nanocomposite particles was added into a 10 ml aqueous solution of tartrazine, which its concentration was 3×10^{-5} M. The pH of all solutions was adjusted at two with HCl as the best pH for photodegradation of tartrazine. The mixture was stirred continuously at 25˚C for 1–5 h under UV-C radiation (CHLIGHTING, T8, 30 w, China), and then centrifuged. Afterwards the concentration of free tartrazine was determined by UV–vis spectrophotometer. The instrument response was periodically checked with the known tartrazine standard solution.

Percentage of degradation was measured by using the Eq. (1) as follows [53]:

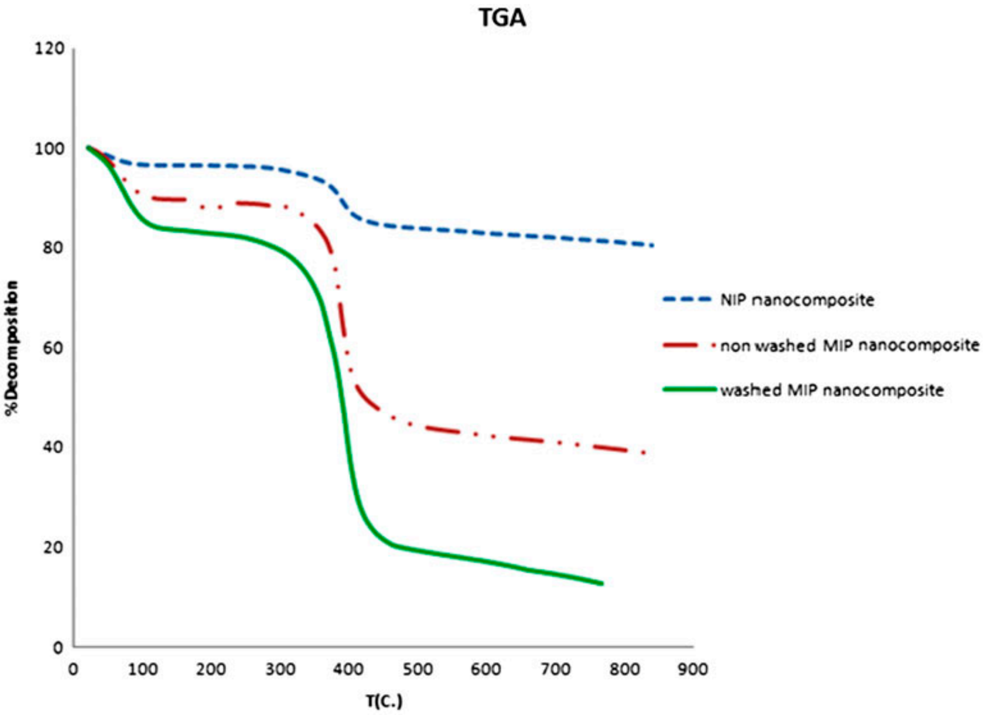

Fig. 3. TGA plots of nanocomposite.

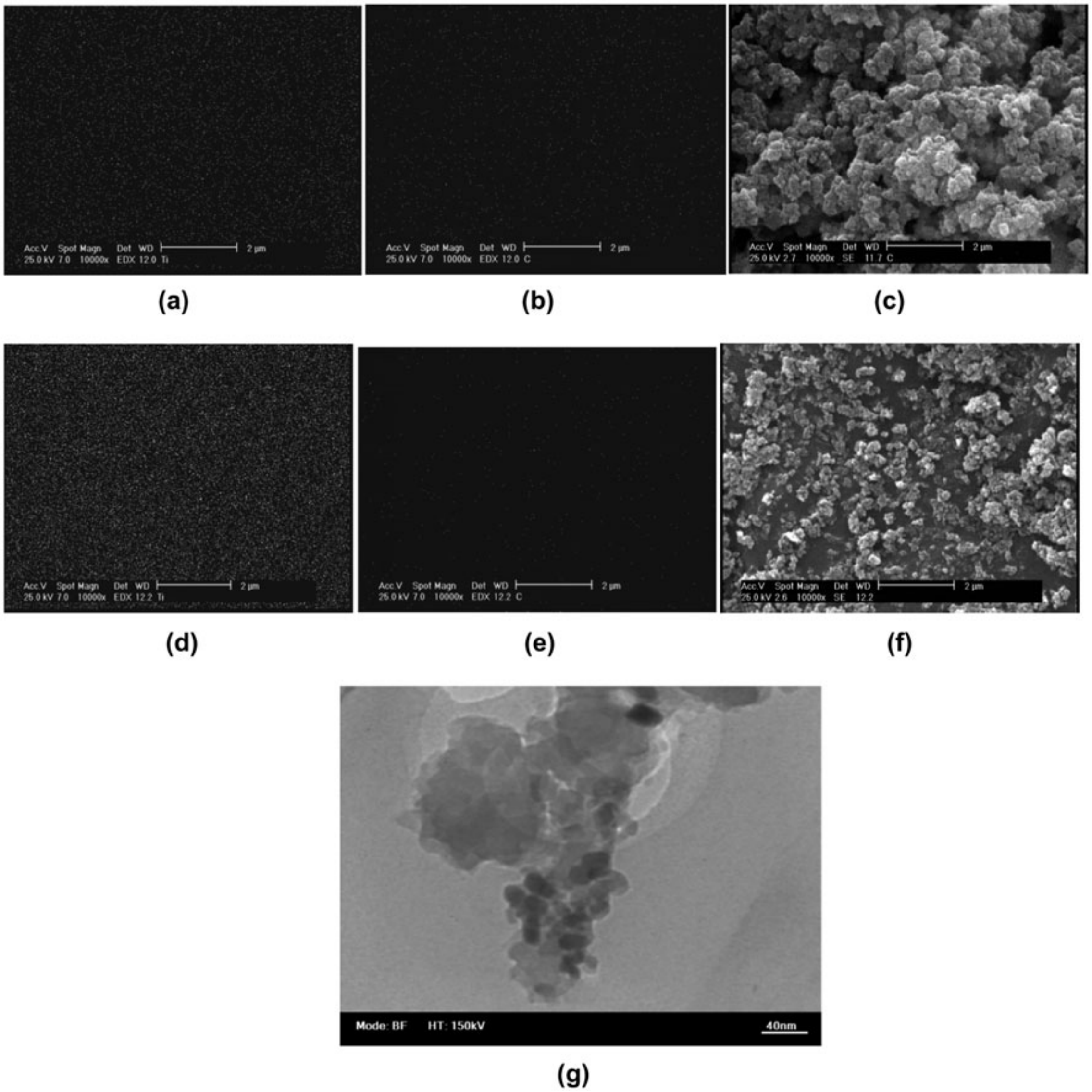

Fig. 4. WDX—Ti (a), WDX—C (b), SEM (c) of MIP nanocomposite and WDX—Ti (d), WDX—C (e), SEM (f) of NIP nanocomposite and TEM of MIP nanocomposite (g).

$$\% \text{ Degradation} = \frac{A_\text{i} - A_\text{f}}{A_\text{i}} \times 100 \qquad (1)$$

where A_i and A_f are the absorbance of tartrazine solution before and after photodegradation process, respectively.

3. Results and discussion

3.1. Characterization

The FT-IR spectra of non-washed and washed MIP nanocomposite and washed NIP nanocomposite were studied at 4,000–400 cm^{-1} (Fig. 2). Similar characteristic peaks indicate the similarity between the backbone structures of the different nanocomposites. A strong peak at ~1,650–1,656 cm^{-1} which was observed in the FT-IR spectra of the washed NIP nanocomposite, non-washed and washed MIP nanocomposite which is attributed to the vibration mode of C=O group. As a result of intermolecular hydrogen bonding with the $-NH_2$ group of AA, the binding vibration at 3,312 cm^{-1} in non-washed MIP nanocomposite were shifted to 3,275 cm^{-1} in the washed MIP nanocomposite and 3,387 cm^{-1} in NIP nanocomposite. A sharp peak at 1,114.54 cm^{-1} shown in non-washed MIP nanocomposite is because of the $-SO_3H$ of tartrazine, which has disappeared in washed MIP nanocomposite

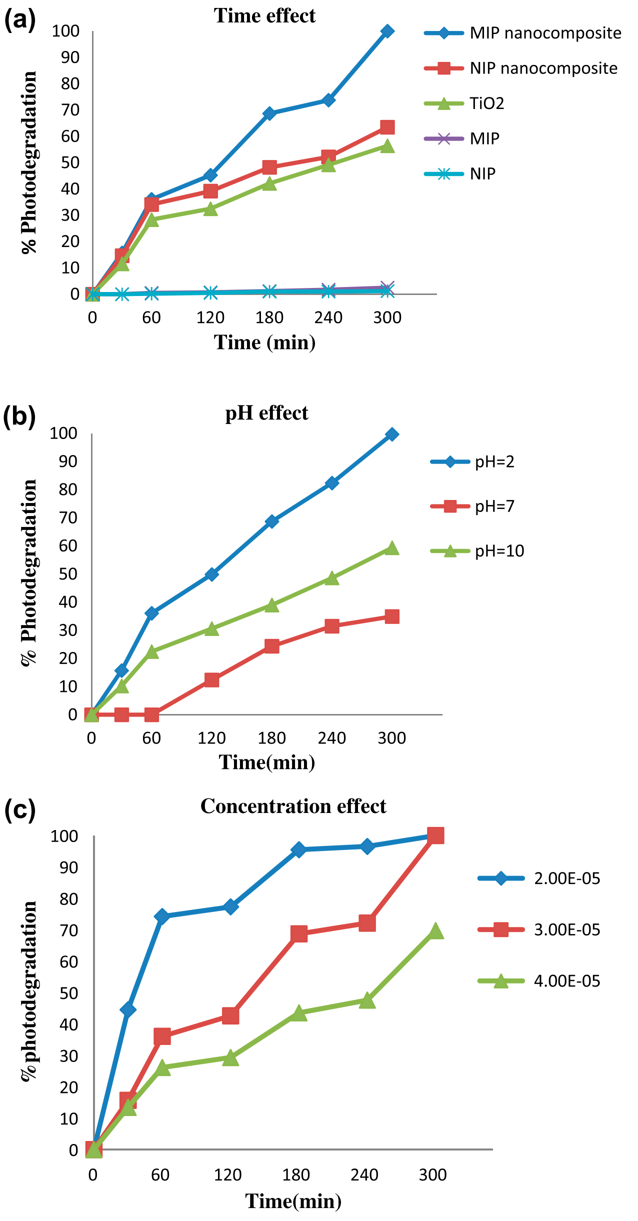

Fig. 5. Effect of time (a), pH (b), and concentration (c) on photodegradation process.

Table 1
The effect of adsorbents type on photodegradation procedure

Type of adsorbents	% photodegradation
MIP	2.5
NIP	1.2
TiO_2	56.43
NIP nanocomposite	63.47
MIP nanocomposite	100

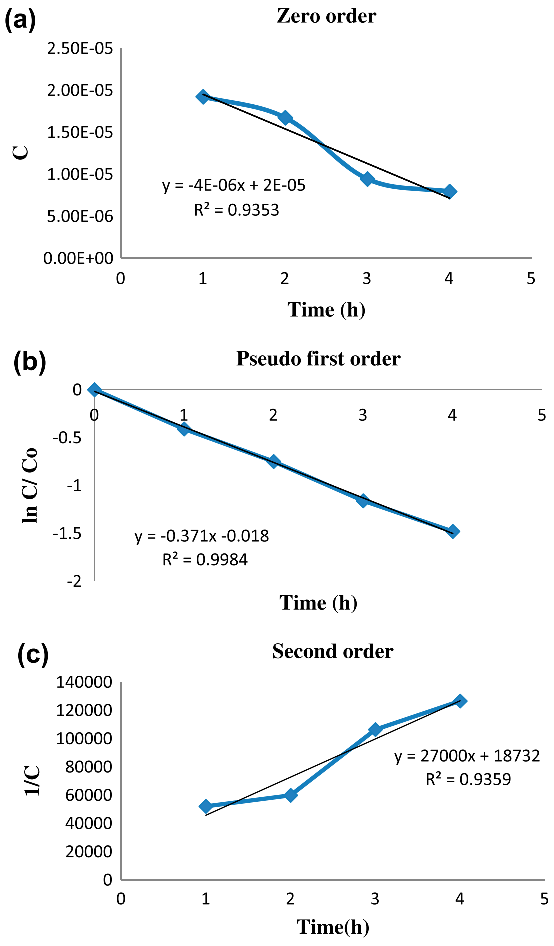

Fig. 6. Kinetic of photodegradation process, (a) zero-order, (b) first-order and (c) second-order reaction.

spectra. The broad spectra in 400–600 cm^{-1} in all nanocomposites are ascribed to TiO_2 particles.

The TGA plot of non-washed and washed MIP nanocomposite and washed NIP nanocomposite is illustrated in Fig. 3. The TGA revealed two decomposition states in non-washed MIP nanocomposite: one mass loses starting at near 85°C is ascribed to the decomposition of free monomer and the other one starting at 250°C is related to the tartrazine decomposition at the melting point. All the organic parts of the nanocomposites were completely decomposed before reaching the temperature to 400°C. One mass loss near 820°C in all nanocomposites is related to TiO_2 decomposition.

Scanning electron microscopy and TEM and X-ray spectroscopy were employed to establish the shape, surface morphology, and particle size of the produced nanocomposite particles. The nanocomposite particles were coated with gold.

As can be seen in Fig. 4(c) porosity of the surface could be clearly observed for the MIP nanocomposite and shows a relatively considerable discrepancy in comparison to NIP nanocomposite which does not possess porous shape (Fig. 4(f)). Fig. 4(g) which is TEM of MIP nanocomposite shows the nanosize of the particles.

As shown in Fig. 4(a), (b), (d), and (e), WDX images studied the map of individual elements and show location and abundance of Ti and C elements as light spots on a black background, which shows a remarkable difference between Ti and C for MIP nanocomposite and NIP nanocomposite.

3.2. Photodegradation of dye

The photodegradation was completed in 5 h time for MIP nanocomposite and reached to 100% then the solution became colorless. As shown in Fig. 5(a), the MIP nanocomposite is more effective than pure TiO_2, NIP nanocomposite, MIP and NIP without TiO_2. The comparison of five adsorbents in photodegradation procedure is revealed (Table 1), the MIP nanocomposite has improved photodegradation of tartrazine by 100% compared to NIP nanocomposite and pure nanophotocatalyst which yielded 63 and 56%, respectively, under the same condition. The TOC analysis revealed that 63.5% of organic carbons were removed.

The pH value has a large influence on the extent of photodegradation of dye from the solution, because it could influence the properties of the sorbent. Photodegradation of tartrazine was investigated at pH values of 2, 7, and 10 (Fig. 5(b)). The percentage value of tartrazine photodegradation increases, when the pH decreases down to 2. So, this pH was used as the best pH for photodegradation of tartrazine in all experiments. The adsorption of tartrazine onto the nanocomposite can be ascribed to the interaction between functional groups of nanocomposite and analyte which depends on pH alterations. The interaction between tartrazine and MIP nanocomposite is caused by hydrogen bonding and electrostatic interaction between undissociated amide groups on nanocomposite surface and sulfonated groups of the dye [52]. The results showed that with increasing the concentration, photodegradation decreases (Fig. 5(c)).

The presence of inorganic salts has complicated influences on the photocatalytic treatment process of the dye [54]. We have studied the effect of NaCl on photodegradation process. In this respect 0.012 g of NaCl was added to 10 ml of a standard solution of tartrazine. The results represented that the salt exhibited the inhibition effect on dye photodegradation. It can be attributed to the scavenging effect of chloride anion on the hydroxyl radical [55].

Fig. 6 shows the R^2 for zero-order, pseudo-first-order, and second-order reaction. The results clearly represent that the tartrazine photodegradation process in water environment follows a pseudo-first-order kinetic ($R^2 = 0.9984$). To carry out the photocatalytic reaction, 0.001 g of nanocomposite was added to the standard solution of tartrazine and put under the UV lamp for 5 h.

4. Conclusion

As it is known removing hazardous tartrazine from the water environment is so important. In this research, high efficiency nanocomposite of nanoMIP-nanophotocatalyst was synthesized for tartrazine. The MIP nanocomposite revealed high affinity to template and photodegrade the color of solution completely under UV radiation. The synthesis of nanocomposite was carried out in water, which is a green solvent. The effects of pH, time, and tartrazine concentration were optimized to remove and photodegrade tartrazine. The results indicated that nanoscale composite (MIP-TiO_2) was an efficient nanocomposite to remove and photodegrade tartrazine from water environment.

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