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PREPARATION AND CHARACTERIZATION OF TITANIUM DIOXIDE NANO PARTICLES USING MICROWAVE DRYING TECHNIQUE

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Abstract

Nano sized TiO_2 was synthesized by hydrolysis of titanium tetra isopropoxide. The prepared TiO_2 nano particles have been structurally characterized by X-ray diffraction (XRD). The energy dispersive spectroscopy (EDX) of the nano particles dispersion confirmed the presence of elemental titanium signal; no peaks of other impurity were detected. The average size and morphology of titanium dioxide nano particles were determined by transmission electron microscopy (TEM). Fourier Transformation Infra Red (FTIR) spectroscopy was used to detect the function groups of organic compounds and to assure the complete solvent removal after heat treatment.

Key Words

Nano sized TiO_2, titanium tetra isopropoxide, anatase, solvent removal, heat treatment.

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1. Introduction

Titanium dioxide (TiO_2) is a nontoxic material and has been applied in environmental treatments such as water and air purification, water disinfection and sterilization because of its unique properties such as strong photocatalytic activity (when irradiated with light having energy higher than the band gap [1].Although TiO_2 is an effective UV absorber and confers significant protection from UV because of rapid attenuation offered by absorption and scattering , the interaction between UV and the TiO_2 surface would generate free radicals ,under UV light ,TiO_2 produces electron-hole pairs. On the basis of this feature, TiO_2 photocatalyst has been successfully employed to purify water and air by degrading organic pollutants. Researches were also carried out to investigate photocatalytic activity of TiO_2 for converting harmful organic waste products into harmless environmentally acceptable materials. As viewed from crystalline structure, TiO_2 is a multi-crystal form substance. It includes brookite, rutile and anatase, although only rutile and anatase are widely used [2].

Both rutile and anatase forms have a square crystal system structure. The crystal density and absorption ability of UV–visible light of rutile are greater than those of anatase, but anatase possesses relatively high reflectivity for UV–Visible light [3].

Nano sized TiO_2 was synthesized by hydrolysis of titanium tetra isopropoxide [4]. Nanosized TiO_2 has been known to be one of the best photocatalysts. The preparation methods for nanosized TiO_2 mainly comprise sol–gel [5]. This study investigates the formation of stable, nanosizedTiO_2 particles via hydrolysis of titanium tetraisopropoxide. Nano-TiO_2 of anatase prepared has been observed to be a type of active, stable and effective photocatalyst after long irradiation time [6]. Sol-gel technique, which includes two steps of sol-preparation and thermal treatment, is a practical method to prepare nano-TiO_2 film [7]. It was found that the crystalloid structure determined by the thermal treating parameters influences the particle size of TiO_2 powder, photo-catalytic and antibacterial performances of the film [8].

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2. Experimental Work

2.1 Materials and preparation method

To prepare 80 ml of TiO_2 sol, 5 mL titanium tetraisopropoxide (TTIP) in 10 mL 2-propanol was added drop-wise to 200 mL solution of deionised water and nitric acid with vigorous stirring and pH was fixed at 1.5. After 24 h, the transparent TiO_2 sol was obtained, and then drying in microwave for 10 min to get TiO_2 nano powder, the drying sample calcinated finally the prepared powder was calcined at 450 °C for 4 h.

2.2 Characterization techniques

The x-ray diffraction (XRD) characterizes the structure of the synthesized nano particles. The peaks in the XRD pattern are in good agreement with the standard values indicating that nanoparticles structure dominantly correspond to anatase crystalline. From the diffraction peaks the crystalline phase was identified as the anatase phase**.** The studies of size, morphology and composition of the nano particles were performed by means of transmission electron microscopy (TEM) and energy dispersive X-ray Analysis (EDX). TEM photographs indicate that the nano powders consist of well dispersed agglomerates of grains with a narrow size distribution (20 and 50 nm), whereas the radius of the individual particles are between 20 and 40 nm.

Fourier Transformation Infra Red (FTIR) spectroscopy is a vibrational spectroscopic technique that uses infrared radiation to vibrate molecular bonds within the sample that absorbs it.

As different samples contain different molecular bonds or different configurations of molecular bonds; FTIR allows one to obtain chemical information on molecules within the sample. The spectra covered the wave number ranging from 4000 to 400cm^{-1}. Special care was taken to prepare the pellets at the same thickness by taking the same amount of sample and applying the same pressure. Therefore, in the present study it is possible to directly relate the intensities of the absorption bands to the concentration of the corresponding functional groups.

2.3 Experiment of Photocatalytic process:

The photocatalytic decomposition of malathion was conducted in a in a 250 ml UV reactor equipped with a 10 W high pressure mercury lamp (mean wavelength 254 nm).

10 mg of the prepared TiO2 powder was added to 100 ml of malathion solution (1gm malathion in 100 ml ethanol) concentration (1000) ppm. Malathion solution was irradiated in the UV reactor. At specific time intervals samples where withdrawn from the reactor and filtered through a 0.7 µm filter then centrifuged to remove the TiO2 particles from the solution, malathion concentrations were determined by GC/FPD.

3. RESULTS AND DISCUSSIONS

3.1. Formation and characterization of TiO_2 nano particles and its properties

Fig.1 shows the XRD pattern of TiO_2 nano particles. The XRD spectrum confirmed the crystalline structure of TiO_2 nano particles. No peaks of other impurity crystalline phases have been detected.

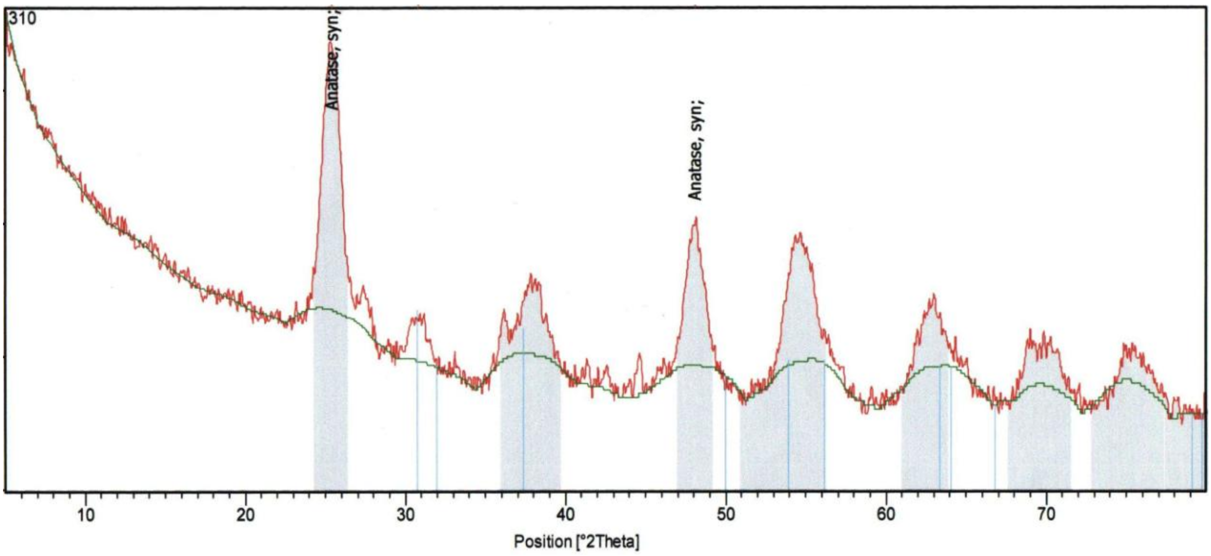

Fig.1. XRD pattern of TiO_2 nano particles

The main peak of θ=25.3° matches the (101) crystallographic plane of anatase of TiO_2 nanoparticles, indicating that nanoparticles structure corresponded to anatase crystalline [9]. Strong diffraction peaks at 2θ values 25.3, 37.8, 48, 55.1, and 64.4 corresponding to the planes of (101), (004), (200), (211), and (220). These peaks are the characteristic peaks of titanium dioxide present on the surface of the substrate. From the diffraction peaks the crystalline phase was identified as the anatase phase [10].

The elemental analysis of the TiO_2 nanoparticles was performed using the EDX on the SEM that shown in Fig. 2 (EDX spectrum) peaks.

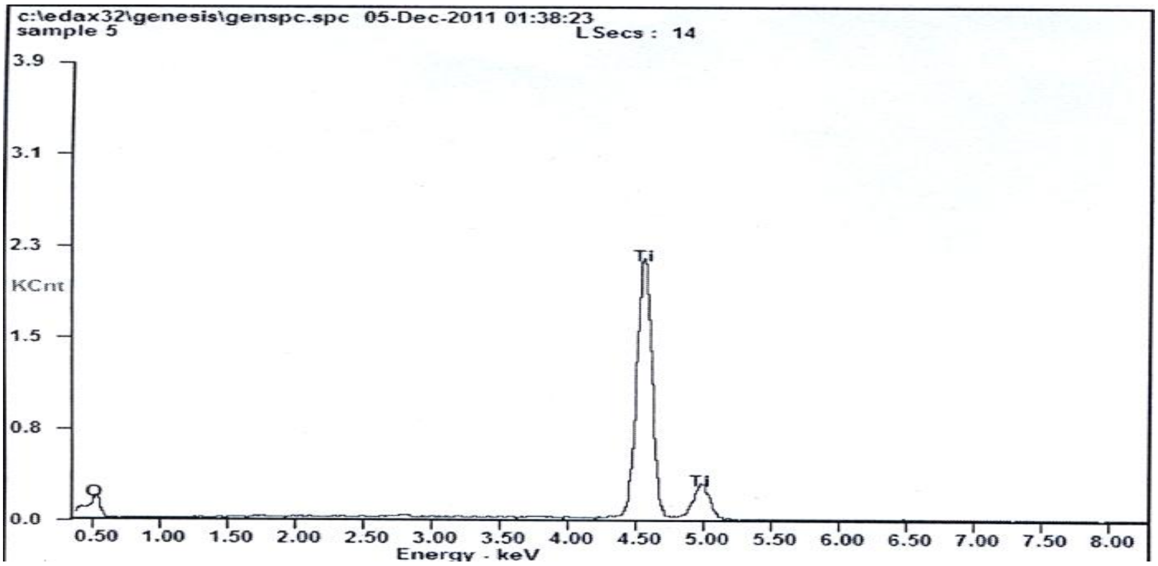

Fig.2. EDX spectrum of TiO_2 nano particles

TEM imaging (Fig.3) shows the agglomerates of small grains and some dispersed nano particles.
The TEM image of the prepared TiO_2 which represented in Fig.3 and indicates well dispersed particles which are more or less spherical. The average size of these particles is typically 20 nm.The particle size histograms of TiO_2 particles (Fig.3) show that the particles range in size from 20 to 60 nm.

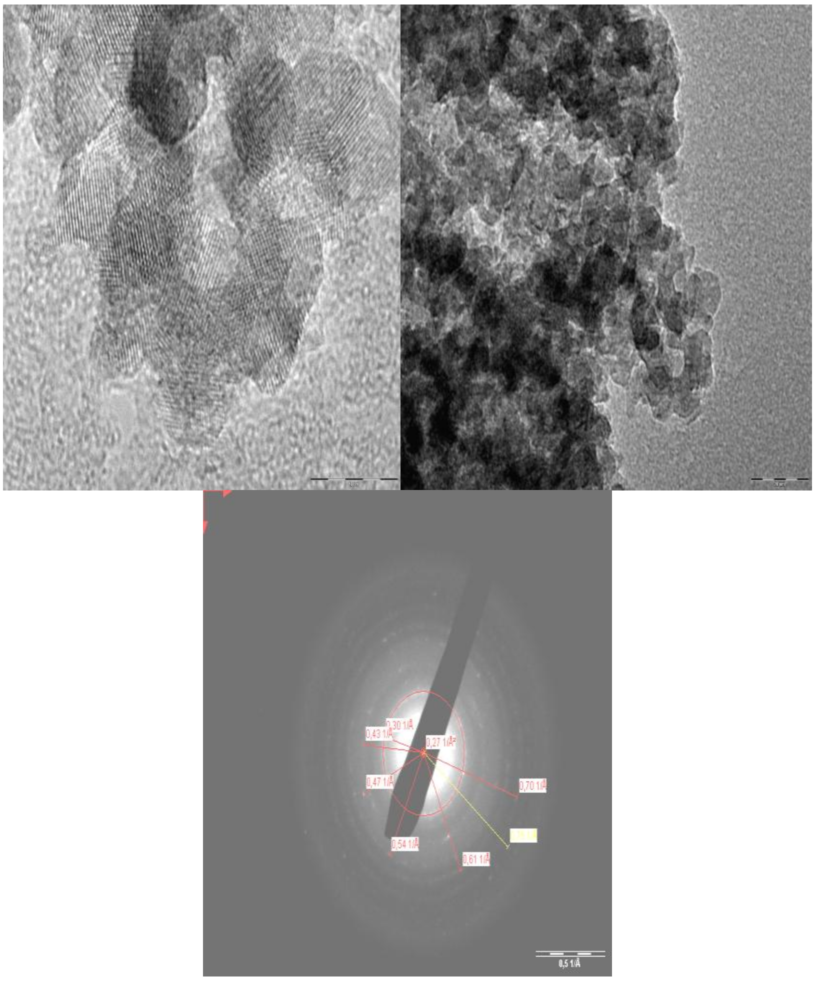

Fig.3. TEM and HEED images of TiO_2 nano particles synthesized

The corresponding High Energy Electron Diffraction (HEED) pattern of TiO_2 particles that shown on the (right-hand in Fig.3) When the electron diffraction is carried out on a limited number of crystals one observes only some spots of diffraction distributed on concentric circles. The rings patterns with plane distances are consistent with the plane families (101), (004), (200), (211), and (220) which represent the pattern of TiO_2 structure [10].
The FTIR spectrum of the TiO2 nanoparticles in Fig. 4 shows the broad peak around 585cm−1 is that for the Ti–O bond in titanium dioxide [11]. The other peaks in the FTIR spectra are due to absorbed organic molecules on the surface of nanoparticles. The broad band around 3000–3500 cm−1 corresponds to the O–H group, while the narrow bands at 2916, 2847, 1618 and 1381 cm−1 are due to organic residues originating from the sample as a result of the preparation procedure [12].

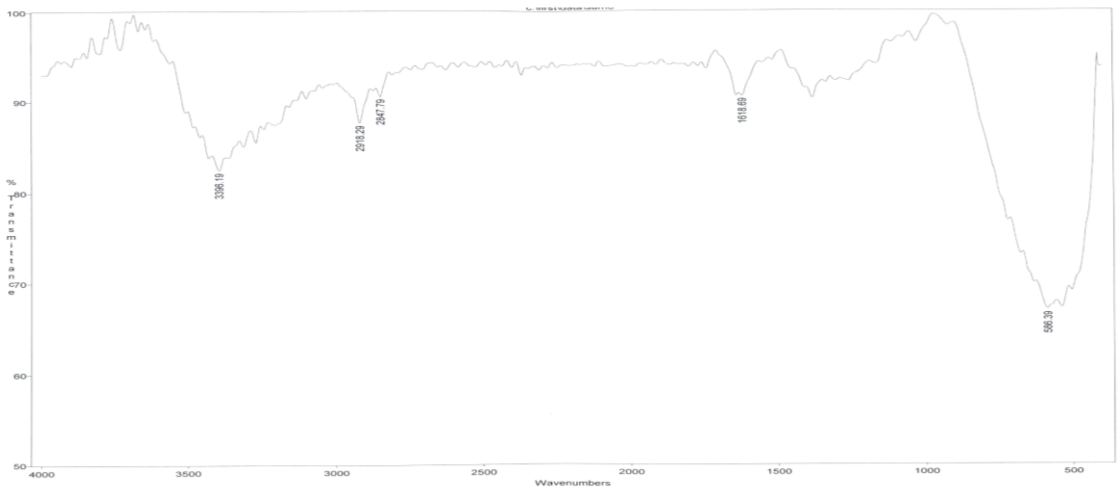

Fig.4. FTIR spectrum of TiO_2 nano particles

3.2. Effect of the prepared TiO_2 nano particles in photodegradation of CWA

The GC/FPD spectrum of malathion shown in Fig. 5 appear at retention time 8.952 min.The peak that appear at 3.857 min illustrate the starting of the degradation of malathion due to storage or decomposition. The peaks that appear at 0.946 min and 0.794 min are impurities.

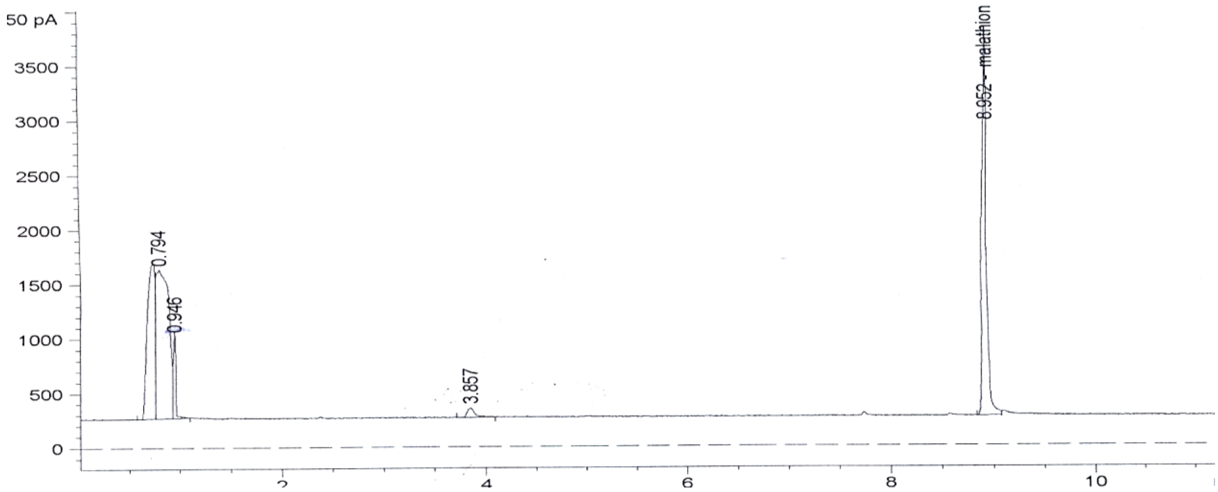

Fig.5. GC/FPD spectrum of malathion

The photodegradation process of malathion during exposure to UV lamp is detected using GC/FPD as shown in Fig.6.The degree of decomposition of malathion varying with time of exposure to UV lamp. The impurities concentrations (at retention time =0.95 and 0.79) seams to be constant during the course of experiment which indicate that they do not produced due to photodegradation. Moreover, the concentration increase (at retention time =3.85) then decrease which indicates that it produce due to self degradation of malathion due to storage.

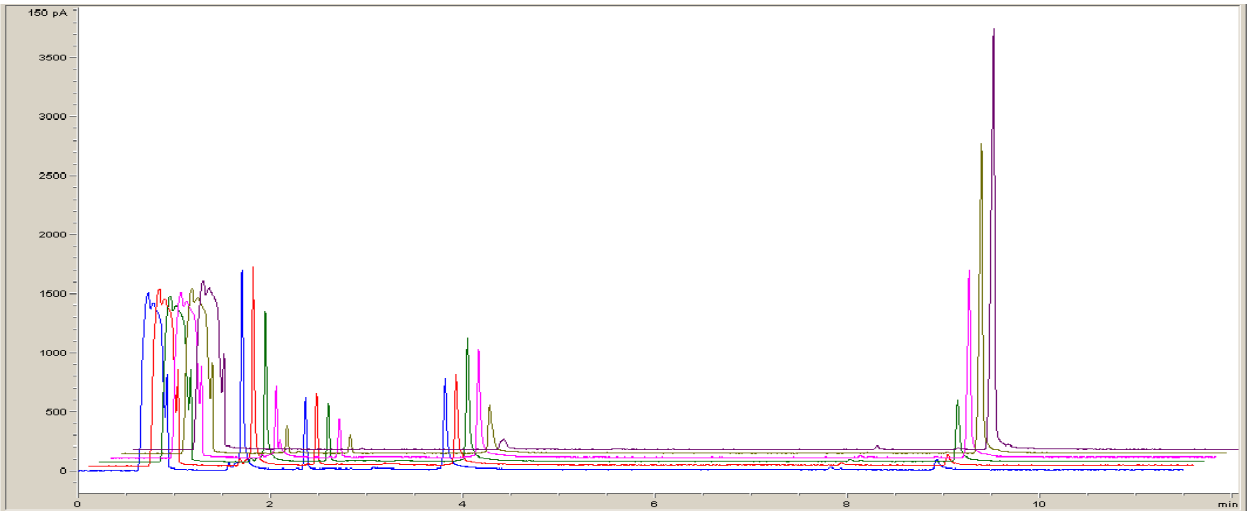

Fig.6. Photodegradation process of malathion during exposure to UV lamp

The photodegradation efficiencies of malathion due to presence of TiO2 is shown in Fig.7. The decomposition increase significantly with an increase in the irradiation time of malathion mixed with TiO2 nano powder. It is clear that, when the initial concentration of malathion was 1000 ppm, the degradation efficiency in 15 min was 68%, while after 30 min, the degradation efficiency was 41%. After 60 min, the degradation efficiency was 18%, after 90 min, the degradation efficiency was 3%, finally after 90 min, the degradation efficiency was 2%.

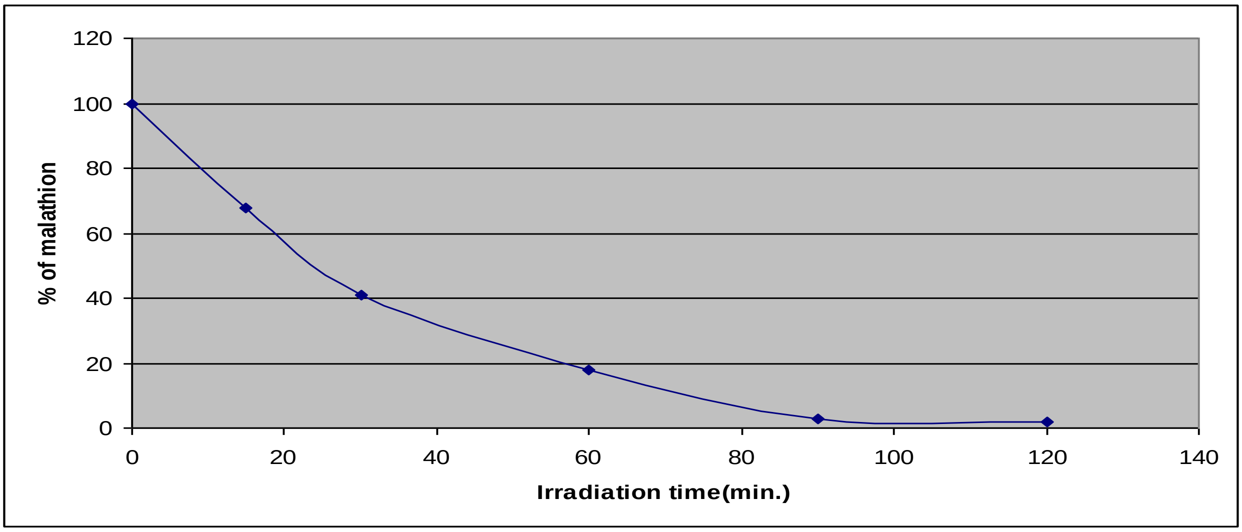

Fig.7. Effect of variation of irradiation time on the photodecontamination process of malathion

4. Conclusion

In summary, TiO2 nanoparticles with mean diameters of 20 nm were synthesized using titanium tetraisopropoxide. The nanoparticles were characterized by FTIR, TEM, EDX and XRD.
FTIR spectra evaluate the function groups of organic compounds and find out the complete solvent removal after heat treatment. The energy-dispersive spectroscopy (EDX) of the nanoparticles dispersion confirmed the presence of TiO2 signal no peaks of other impurity were detected. TEM illustrate the average size and morphology of titanium dioxide nano particles.
XRD illustrate the crystalline structure of the synthesized TiO2 nano particles. HEED confirmed the formation of TiO2 nanoparticles. Additionally, the effect of TiO_2 nano particles of anatase in the photo degradation of CWA was appearing by analysis of the sample by GC-Ms and GC/FPD.
The results of this study clearly demonstrated that the synthesized TiO_2 nanoparticles used in photodegradation of malathion that the degradation efficiency increase with increasing the irradiation time to UV lamp.

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