TiO$_2$/graphene nanocomposite for photocatalytic application

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TiO$_2$ nanomaterial, including nanoparticles (NP) and nanowires (NW), has been intensely investigated as photocatalyst for water purification and environment decontamination, while the photon generated electron and hole pair (EHP) recombination is one of factors limiting its efficiency. TiO$_2$/graphene nanocomposite is very promising to overcome this limitation for photocatalytic applications. Graphene, with its unique electronic properties, large specific surface area and high transparency, contributes to facile charge separation and adsorptivity in this hybrid structure. In this chapter, we summarize our recent work on the development of TiO$_2$/graphene nanocomposite photocatalysts; especially we compared the difference of graphene-TiO$_2$ nanoparticle (GNP) and graphene-TiO$_2$ nanowire (GNW) composites [1]. The structural, chemical, electrical, and optical properties of the composite materials were studied, and their photocatalytic performance was evaluated based on photo degradation of methylene blue under visible light. We demonstrated that uniform dispersion of TiO$_2$ on graphene is critical for the photocatalytic effect of the composite. It was found that by incorporation of graphene, GNP and GNW have higher performance than their counterparts of pure TiO$_2$ NP and NW. Furthermore, it was found that NWs have more uniform distribution on graphene with less agglomeration, resulting in more direct contact between TiO$_2$ and graphene, and hence further improved EHPs separation and transportation. The adsorptivity of GNW is also significantly higher than GNP. The result is that photocatalytic activity of GNW is much higher than GNP and pure NWs or NPs.

Keywords: Photocatalyst; TiO$_2$; graphene; nanocomposite; nanowire

1. Background

1.1 TiO$_2$ as photocatalyst for decontamination

Photocatalyst, generally speaking, is a catalyst that participates and modifies the reaction rate of chemical reactions under light irradiation without changing and consuming itself in the end. Since the discovery that TiO$_2$ particles were capable of trapping and oxidizing organic compounds to minerals and small molecules such as CO$_2$, extensive studies have been conducted to explore the potential of TiO$_2$ as photocatalyst for environmental decontamination such as water purification and air cleaning. As shown in Fig. 1, TiO$_2$ semiconductor absorbs UV photons from sunlight generating electron and hole pairs (EHPs). When the hole reaches the particle’s surface, it can react with hydroxyl ions from adsorbed surface water and form highly reactive hydroxyl radicals that are electrically neutral but highly reactive. Airborne or water pollutant molecules can be adsorbed on the TiO$_2$ particle surface and react with adsorbed hydroxyl radicals, converting into minerals and small molecules. Such a photocatalytic degradation process applied for environmental decontamination has several advantages: (1) the material used is environment-benign and easy to be separated for recyclable usage; (2) the reaction process is simple and can take place under ambient conditions; (3) many different kinds of pollutants in wastewater or in air can be oxidized simultaneously within a short time. Therefore, photocatalytic degradation (PCD) of contaminants using TiO$_2$ as photocatalyst is being under study for disinfection, air purification, environmental cleaning, and wastewater treatment in daily life and industrial activities [2].

![Fig. 1 Schematic representation of the photocatalytic degradation of pollutants at TiO$_2$ surface. After UV photons absorption, EHPs are generated with holes as the stored energy that can react with hydroxyl ions (OH) from adsorbed surface water and form highly reactive hydroxyl radicals (OH). When pollutant molecules are adsorbed on TiO$_2$ particle surface, they react with these hydroxyl radicals into minerals and CO$_2$. The photoelectrons in TiO$_2$ can transfer to O$_2$ or others to restore its charge neutrality.](image-url)
1.2 Motivation of using TiO$_2$/graphene composite

Graphene is promising for energy and environment related applications [3], such as energy storage [4], photovoltaics [5], photoelectrochemical [6] and photocatalytic [7-9] based hydrogen/hydrocarbon fuel generation, and photocatalytic degradation of organic contaminants. So far, carbon-related materials have been extensively studied [10] for catalytic applications either by serving as a supporting matrix to tailor the electronic or photonic properties of catalysts, or as catalyst by itself. Graphene, the single atomic layer of graphite, with its very high electric charge carrier mobility and optical transparency, intrinsic large surface and capability of chemical functionalization, and mechanical flexibility and strength, could be an ideal mechanical support and charge carrier shuttle of photo sensitizers and catalysts to construct nanocomposites with enhanced performance based on the synergistic effects of components. Furthermore, unlike electronic and photonic applications which call for high purity sp$^2$ bonded graphene that can only be produced by delicate approaches, energy and environment related applications require large-quantity and cost-effective methods to produce surface-functionalized and single-layer or few-layer graphene, which fortunately can be easily produced through the well-known Hummer’s method [11]. The resulted single or few layer graphene oxide (GO) from this method contains abundant hydrophilic groups such as hydroxyl, carboxyl, and epoxyl groups bonded with carbon and other atomic scale defects. They significantly distort sp$^2$ in-plane bonding and act as scattering centers that dramatically alter the electrical and optical properties of graphene with low transparency, high resistivity and low charge carrier mobility. Repairing the sp$^2$ aromatic structure and reduction of GO to the so-called reduced graphene oxide (RGO) is necessary to be used for charge shuttling and possible catalytic sites. Chemical reduction using reducing agent such as hydrazine [12], photochemical reduction using semiconductors [13], and hydrothermal reduction using supercritical water [14] have been demonstrated to be effective for this purpose. The latter two “green” routes that simultaneously reduce GO and anchor semiconductor nanoparticles (NPs) on RGO are more intriguing.

With its 3.2 eV electronic band gap, TiO$_2$ is only sensitive to the light wavelengths below ~ 380 nm that belong to the UV range. Nevertheless, the cost-effective TiO$_2$-based materials, because of their high oxidation capability and extreme chemical stability against the strong oxidation environment in photocatalytic applications, have been the most popular photocatalysts [15]. To facilitate the use of TiO$_2$ powder as photocatalyst, immobilization on a suitable substrate is desired. Carbon-based materials of different origin, including activated carbon, carbon black, carbon fiber, and carbon nanotubes (CNTs), have been investigated for this purpose [16, 17]. As a new member of carbon family, graphene, due to its superior properties mentioned above, used as mechanical support and electric shuttle in composites for anchoring TiO$_2$ NPs has also been reported [6, 8, 14, 18-24].

Instead of uniformly distributed on graphene, the loaded NPs easily form chemical bonds and accumulate along the wrinkles of graphene sheets or other defects and form agglomerations. In comparison with pure TiO$_2$, the enhanced photocatalytic effect of CNT-TiO$_2$ [25] and graphene-TiO$_2$ composites was generally attributed to three factors, including enhanced EHP separation for high quantum efficiency, extended visible light absorption for visible light harvesting, and dramatically boosted reactant adsorbability giving high reaction possibility [1]. The heterojunction between graphene and TiO$_2$ promotes separation of EHP in TiO$_2$ with electron injecting into graphene that acts as electron sink hindering recombination, while the hole remaining in TiO$_2$ to drive the oxidation process. The formation of Ti-O-C bonding by using carbon as dopant in TiO$_2$ to extend TiO$_2$ light absorption cutoff wavelength was also considered as one extra benefit. Unfortunately, the agglomeration of TiO$_2$ NPs on graphene prohibits the direct chemical contact between the two components and therefore will dramatically diminish these two benefits. The large surface area to easily catch the target for photo degradation, another merit of graphene, has to be discounted if TiO$_2$ is not uniformly distributed, since the holes in TiO$_2$ cannot reach those areas to oxidize the adsorbed targets. To exploit the benefits of synergistic photocatalytic effects of TiO$_2$ and graphene other than using graphene simply as a mechanical support, dispersion of TiO$_2$ on graphene with minimal agglomeration is critical.

Here we report our study on the synthesis and property of the composite of TiO$_2$ nanowires (NWs) grafted onto graphene, called as GNW here. In comparison with TiO$_2$ NPs on graphene (GNP) structure, GNW composite is not a simple morphology change of TiO$_2$ from NP to NW. The most prominent merit to use NWs other than NPs is that NWs have a large opportunity than NPs to be uniformly grafted onto graphene to form bonding simply based on geometric consideration. Therefore, the photoelectron injection into graphene will be much easier to occur, with the potential for higher photocatalytic efficiency. The superior photocatalytic ability of GNW over GNP was confirmed in this study.

2. Synthesis process

2.1 Synthesis of TiO$_2$ nanowires (NW)

Hydrothermal approach was adopted to synthesize crystalline anatase TiO$_2$ NWs. In a typical preparation procedure, 3 g TiO$_2$ Degussa P25 powders (EVONIK Industries) were added into 100 mL KOH aqueous solution (10 M) and stirred for 30 min until a homogeneous suspension was gained. A Teflon-lined autoclave (125 mL capacity) was filled with this suspension up to 80% of the total volume. Sealed into a stainless steel tank, the autoclave was kept at 200°C for 24 hours and then cooled down naturally to room temperature. The obtained product was successively washed with dilute
HCl aqueous solution, deionized water and methanol for several times until the pH value equaled to 7. After recovered by vacuum filtration, the wet product was baked at 70°C for 6 hours, and eventually the white-color anatase TiO₂ NW powder was obtained.

2.2 Synthesis of graphene oxide
Expanded flake graphite (3805, donated by Asbury Graphite Mills, Inc.) was used for preparation of graphene oxide. The expanded graphite was first oxidized into graphite oxide via modified Hummers’ method. In detail, 3 g of graphite was added into a mixture of 2.5 g K₂S₂O₈, 2.5 g P₂O₅, and 12 mL concentrated H₂SO₄. After heated to 80°C and kept stirring for 5 hours, 500 mL deionized water was slowly added into the mixture for dilution. Reoxidization was implemented by the addition of a large amount of deionized water (500 mL) and treated with 30% H₂O₂ solution (10 mL). By filtration through 0.22 μm Nylon film repeatedly with deionized water until the pH of the filtrate was neutral, the graphite oxide was obtained after drying the product in oven at 60°C for 2 hours. Exfoliation was performed by sonicating 0.1 mg/mL of graphite oxide dispersion for 1 hour. The graphene oxide (GO) was recovered by filtration again and vacuum drying.

2.3 Synthesis of graphene-TiO₂ composites
Hydrothermal method was employed to synthesize graphene-TiO₂ nanowire GNW nanocomposite. 30 mg of GO was mixed into a solution of 90 mL deionized water and 30 mL ethanol under sonication for 1 hour to re-exfoliate the GO thoroughly, and 3 g as-made TiO₂ NWs was added to the GO suspension. Then the sonication and stirring was applied alternately for 2 hours with 30 min for each step until a homogeneous suspension was achieved, which shows a uniform light gray color. The suspension was then poured into a Teflon-lined autoclave of 125 mL capacity and maintained at 120°C for 3 hours to synthesize GNW composite. This process simultaneously reduces GO to RGO by electron donation from ethanol and formed Ti-O-C bonding between TiO₂ and RGO. After cooled down to the room temperature, the suspension was filtered several times with deionized water and the product was dried at ambient condition. For direct comparison, GNP nanocomposite was also prepared according to the same procedure described above, in which the TiO₂ NWs were replaced with TiO₂ P25 NPs.

3. Material Characterization

3.1 Electron microscopy
Both scanning electron microscope (SEM) and transmission electron microscope (TEM) were utilized to characterize the morphologies of RGO and the as-synthesized TiO₂ NWs, GNP and GNW composites. Figure 2 (a) shows a typical SEM image of the as-made TiO₂ NWs, which was pretty tidy without any contaminant on their surface. The NW structure was observed to be bunches of NWs with several microns long, but the width varies from about 20 nm up to 200 nm, namely nanosheets. Further sonication will break down these nanosheets into single NWs with diameter of about 20 nm as shown in Fig. (a) inset. We also used the hydrothermal method to reduce GO without adding TiO₂ nanostructures for control experiment, and the TEM image of RGO structure in Fig. 2 (d) demonstrates that this reduction process with ethanol as the reductive agent is effective to produce graphene sheets with thin layers. After a second hydrothermal process, the representative TEM images of GNW nanocomposite structure with different magnifications were shown in Fig. 2 (b) and (c), which presented a uniform covering of TiO₂ NWs over graphene sheets. By comparing Fig. 2 (b) with (a), it is noticed that the wide titanate nanosheets decomposed into very thin NWs that cover RGO surface in a reasonable uniform fashion. The SEM image of as-synthesized GNP nanostructure was shown in Fig. 2 (e), which suggests that the dense TiO₂ NP attachment was scattered over through the graphene sheets on both sides. In contrast with GNW, TEM image in Fig. 2 (f) show that in GNP nanocomposite structure, NPs are mainly attached on the ridges and wrinkles of RGO to form agglomeration, indicating that TiO₂ is easier to form Ti-O-C bond with RGO at the locations of defects, including functional groups. After initial anchoring of a few NPs at these locations, other NPs prefer to form agglomeration around these spots. In contrast, NWs with their significant length, find more opportunities to anchor directly on RGO with less agglomeration.

3.2 X-ray diffraction (XRD)
X-ray diffraction was employed to analyze the crystalline phase of TiO₂ NP, NW, GNP and GNW. The XRD pattern of NP is almost identical to that of GNP which also happened to the NW and GNW group. Only the XRD patterns of GNP and GNW are shown in Fig. 3. It suggests that the hydrothermal process for composite synthesis has no obvious impact on the original TiO₂ crystallization. It is noticed that no diffraction patterns from carbon species could be observed, which may result from the small amount and weak intensity of graphene. The GNP showed almost identical XRD patterns to the pure TiO₂ P25 NP with the typical diffraction peak (101) of anatase phase along with other peaks of (004), (200), (105), etc. and a rutile peak (110) along with other relatively weak peaks of (101), (111), (211) and (220).
In addition, it is worth noting that there is no rutile peaks in GNW detected as that shown in GNP patterns, indicating that TiO$_2$ NW synthesized by the hydrothermal method described in section 2.1 is in pure anatase phase.

![Fig. 2](image)

**Fig. 2** (a) SEM image of TiO$_2$ NWs made through hydrothermal method with KOH and TiO$_2$ P25 with the inset of TEM image; (b) TEM image of the graphene-TiO$_2$ nanowire (GNW) composite; (c) magnified TEM image of (b); (d) TEM image of reduced graphene oxide (RGO); SEM image (e) and TEM image (f) of the as-synthesized graphene-TiO$_2$ nanoparticle (GNP) composite.

![Fig. 3](image)

**Fig. 3** Representative XRD spectra of graphene-TiO$_2$ nanoparticle (GNP) and graphene-TiO$_2$ nanowire (GNW) nanocomposites. A and R refers to anatase and rutile phases, respectively.

### 3.3 Diffuse reflectance absorption spectroscopy

UV-visible diffuse reflectance spectra of NP, NW, GNP and GNW are shown in Fig. 4. The sharp absorption edge was observed at around 400 nm for bare TiO$_2$ as expected, while the increased absorption in the visible light region were detected for both GNP and GNW nanocomposites as a result of the introduction of graphene. And it is notable that compared to bare TiO$_2$, NP and NW, the absorption edge of GNP and GNW underwent a red-shift of about 20 nm and 10 nm respectively. This is attributed to the formation of chemical bonds of Ti-O-C between TiO$_2$ and graphene. Not shown here, the Fourier transform infrared spectroscopy (FTIR) transmission spectra also indicate the formation of Ti-O-C chemical bonds.
3.4 Raman spectroscopy

Raman spectra of NP, NW, GNP and GNW are displayed in Fig. 5. The Raman spectra of GNP and GNW show the typical features of RGO with the presence of D band located at 1340 cm\(^{-1}\) and G band at 1581 cm\(^{-1}\). G band provides information on the in-plane vibration of sp\(^2\) bonded carbon atoms [26], while the D band was attributed to the presence of sp\(^3\) bond and other defects in graphene [27]. The 2D band at around 2780 cm\(^{-1}\) originated from two phonon double resonance. Raman scattering shows a symmetric peak of the 2D band, which is different from that of graphite fitted with two peaks [28]. This demonstrates that the graphene has been successfully exfoliated and incorporated into the composite. The Raman lines for \(E_{2g}\), \(B_{1g}\), \(A_{1g}\) or \(B_{1g}\) modes of TiO\(_2\) anatase phase were also observed.

4. Electrochemical Study and Photocatalytic Performance

4.1 Cyclic voltammetry (CV)

The paste was made by using acetoneacetyl and deionized water as solvent to disperse powders of NP, NW, GNP and GNW with a few drop of Triton X-100. The working electrodes for electrochemical testing were made by depositing the as-made paste on conducting fluorine-doped SnO\(_2\) glass substrate (FTO, TEC 15) via doctor blade method. The working electrodes with an area of 1×1 cm\(^2\) and a film thickness around 10 µm, measured by a profilometer, were calcinated at 450°C for 2 hours in Ar atmosphere.

Cyclic voltammetry (CV) experiments were carried out in a three-electrode cell with SCE as the reference electrode and Pt wire as the counter electrode at a scan rate of 50 mV/s. The electrolyte was a mixture of 2.5 mM K\(_3\)[Fe(CN)\(_6\)] /K\(_4\)[Fe(CN)\(_6\)] with 1:1 mol ratio in 0.1 M KCl solution. Figure 6 shows clear anodic and cathodic peaks for each sample. The peak at positive potentials on the anodic (forward) sweep around 0.3 V vs. SCE represents the oxidation of ferrocyanide to ferricyanide with the loss of one electron. GNW and NW presented the smallest peak-to-peak
separations ($\Delta Ep$) indicating highly improved reaction reversibility. Furthermore, the composites of GNW and GNP displayed large current density. In fact, the anodic current density for GNW exhibits an enhancement of 2.4 fold compared to that of the bare NP surface, demonstrating a significantly enhanced rate of electron transfer attributed to the induction of the graphene as a highly conducting substrate. In addition, the NW, GNP, and GNW all show an improvement in electron transfer over the bare NP surface, which comparably exhibits sluggish kinetics.

![Cyclic voltammograms of NP, NW, GNP and GNW.](image)

**Fig. 6** Cyclic voltammograms of NP, NW, GNP and GNW.

![Nyquist plots of the EIS data of the as-made thin films on FTO glass with NP, NW, GNP and GNW.](image)

**Fig. 7** Nyquist plots of the EIS data of the as-made thin films on FTO glass with NP, NW, GNP and GNW.

### 4.2 Electrochemical impedance spectroscopy (EIS)

One fundamental assumption to use TiO$_2$-graphene hybrid structure is that the heterojunction between TiO$_2$ and graphene will enhance the photogenerated EHPs separation with electrons from the conduction band of TiO$_2$ injected into graphene, while the hole trapped in TiO$_2$ will have longer lifetime to diffuse to the surface for oxidation reaction. Since the photocatalytic process involve the oxidation process by the hole transfer from photocatalysis to the degradable chemical in solution, and reduction process by electron transfer from the photocatalyst to the solution, photocarriers migration in the photocatalytic solids, through the solid-solid junction, and across the solid-liquid junction will determine the reaction kinetics and rate. High conductivity of graphene renders it as a super charge-carrier transport medium, and the large surface area may also result in higher carrier transfer rate between the photocatalyst and the solution/photodegradable chemicals. Electrochemical impedance spectroscopy (EIS) is a very useful tool to characterize the charge-carrier migration. To shed light on the charge transport performance of the different photocatalysts, EIS characterization was carried out for NP, NW, GNP and GNW based electrodes in electrolyte containing I$^-$/I$_3^-$ as the redox couple. Figure 7 shows the EIS measurement in Nyquist plots. Both the nanocomposites of GNP and GNW showed depressed semicircles at high frequencies comparing with their pure TiO$_2$ counterparts. The reduced semicircles indicate diminished resistance of working electrodes, suggesting a decrease in the solid state interface layer resistance and the charge transfer resistance across the solid-liquid junction on the surface by forming hybrid structures of TiO$_2$ with graphene [29]. In addition, the NW structure could also provide a direct path for excited electrons and suppressed the carrier scattering which is a common phenomenon in a NP system. By comparison in Fig. 7, NW systems are superior to NP systems with shorter semicircles suggesting a rapid transport of charge carriers and an effective charge separation.

### 4.3 Photocatalytic study

Photocatalytic activity of each synthesized material was investigated by evaluating the photodegradation rate of methylene blue (MB) under solar illumination (an AM 1.5 solar simulator, Photo Emission Tech SS 50B+) with 100 mW/cm$^2$ intensity. For each measurement, 25 mL of 2 ppm MB solution was prepared and 25 mg of photocatalyst was added. One thing that must be taken into consideration is the evaporation loss during the illumination which was balanced by adding the equal distilled water, and then the absorbance was measured at 663 nm. The remaining MB concentration of each photocatalyst was plotted as a function of irradiation time by using Beer-Lamberts Law, as displayed in Fig. 8 (a). The changes of normalized concentration ($C/C_0$) of MB with irradiation were assumed to be proportional to the normalized maximum absorbance ($A/A_0$). It was obvious that the photodegradation activity of GNW and GNP nanocomposites exhibited significant improvements compared to bare NP and NW. An initial sharp drop of MB concentration was exhibited at the first 10 min for GNW, which could be largely attributed to the strong
absorptivity of GNW. It is known that the decrease of MB concentration in solution results from the physical adsorption on the photocatalyst surface and the chemical decomposition of the absorbed MB. To study the influence simply resulting from adsorption, a parallel control experiment was conducted for these four photocatalysts. The same 2 ppm MB solutions with 25 mg photocatalysts in them were kept in dark environment for 80 min that is long enough for a complete adsorption of MB on the photocatalyst surface as a reference point. Then the remaining concentration fraction of MB in the solution was displayed as column plot in Fig. 8 (b). Apparently, after equilibrium in the dark, GNW presented an excellent property of adsorptivity which is crucial for an efficient photocatalyst. And worthy noting, the adsorptivity for GNW and GNP should not only originate from simple physical adsorption, but also from the \( \pi \)-\( \pi \) stacking between MB and aromatic regions of RGO which has giant \( \pi \)-conjugation system and 2D planar structure. By comparing NP with NW in Fig. 8 (b), the NW structure was also demonstrated to be contributed to trapping contaminants. Consequently, combining the both advantages, GNW exhibited the highest photocatalytic activity.

Fig. 8 (a) Photodegradation of MB under solar light, where \( C_0 \) is the initial concentration of MB and \( C \) is the concentration of MB after irradiation of the sample in the corresponding time interval. (b) Column plot showing the remaining MB in solution after kept in dark for 80 min until equilibrium of the MB concentration.

It is worth to emphasize that in Fig. 8 (a), the dramatic drop of MB concentration after 10 minutes light radiation with GNW or GNP as photocatalyst is caused by the synergetic effects of absorption and degradation. This can be easily identified by the observation that MB concentration is \( \sim 20\% \) at absorption equilibrium, while it is only \( \sim 2\% \) after 10 minutes light irradiation. Comparing graphene-TiO\(_2\) composite structure with pure TiO\(_2\), the enhanced photocatalytic activity can be attributed to the significantly enhanced EHPs separation with the electron injection into graphene to prohibit the photogenerated EHPs recombination, and the remaining hole in TiO\(_2\) will trap at surface state to cause oxidation of MB. In this oxidation process, absorption may also indirectly enhance the reaction rate for hole transfer based on the mechanism proposed by Zhang et al [30]. In this scenario, the improved photocatalytic activity of GNW over GNP can be explained based on the reaction mechanism in addition to the enhanced absorption. As confirmed by the TEM observation in Fig. 2, TiO\(_2\) NWs exhibit more uniform distribution on RGO than NPs, so more NWs have direct chemical bonding (electric contact) with RGO while most NPs do not have. Further considering that carrier transport in a straight path in a NW, in contrast with the zigzag path in agglomerated NPs, electron injection from NW to RGO will be much easier than that in agglomerated NPs. The result is that the EHPs recombination rate in GNW will be much slower than in GNP. Moreover, the strong absorption of MB on GNW also leads to more opportunity of MB molecules close to holes for oxidation. The synergetic effect results in a significant higher photocatalytic capability of GNW over GNP.

5. Conclusion

In conclusion, the graphene-TiO\(_2\) nanowire composite has been successfully synthesized via hydrothermal method. GNW displayed a more uniform dispersion of NWs over graphene sheet over that of NPs on GNP structure. Based on comparative studies of NP, NW, GNP and GNW, it was found that the nanocomposites present excellent adsorptivity of contaminants and high rate of contaminant decomposition. Moreover, a significant improvement of graphene-TiO\(_2\) nanowire composite over other materials in the catalytic photodegradation under solar light has been demonstrated, which is attributed to the enhanced charge separation and transportation by inducing graphene. The results indicated that graphene-TiO\(_2\) nanowire composite is a promising photocatalyst for the remedy of various environmental problems such as water decontamination and air cleaning.


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