Research Article

Enhancement of Visible-Light Photocatalytic Activity of Mesoporous Au-TiO$_2$ Nanocomposites by Surface Plasmon Resonance

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Mesoporous Au-TiO$_2$ nanocomposite plasmonic photocatalyst with visible-light photoactivity was prepared by a simple spray hydrolytic method using photoreduction technique at 90°C. The prepared samples were characterized by X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and N$_2$ adsorption-desorption isotherms. The formation of hydroxyl radicals ($\cdot$OH) on the surface of visible-light illuminated Au-TiO$_2$ nanocomposites was detected by the luminescence technique using terephthalic acid as probe molecules. The photocatalytic activity was evaluated by photocatalytic decolorization of Rhodamine-B (RhB) aqueous solution under visible-light irradiation ($\lambda > 420$ nm). The results revealed that the TiO$_2$ could be crystallized via spray hydrolysis method, and the photoreduction technique was facilitated to prepare Au nanoparticles in the mesoporous TiO$_2$ at 90°C. The light absorption, the formation rate of hydroxyl radicals, and photocatalytic decolorization of Rhodamine-B aqueous solution were significantly enhanced by those embedded Au nanoparticles in the Au-TiO$_2$ nanocomposites. The prepared Au-TiO$_2$ nanocomposites exhibit a highly visible-light photocatalytic activity for photocatalytic degradation of RhB in water, and their photocatalytic activity is higher than that of the pristine TiO$_2$ nanoparticles due to the surface plasmon resonance.

1. Introduction

Since the discovery of photocatalytic water splitting on TiO$_2$ single-crystal electrodes by Fujishima and Honda in 1972 [1], nanosized TiO$_2$ semiconductor has been always regarded as one of the most promising photocatalysts in practical applications, especially water cleaning and removal of volatile organic compounds (VOCs) in air, due to its high photocatalytic activity, chemical stability, low cost, and nontoxicity [2–12]. Recently, highly dispersed noble metal nanoparticles with desirable morphologies have attracted great attention due to their unusual catalytic, electric, optical properties and surface plasmon absorption, and their widespread potential usefulness in diverse fields such as biomedicine, photocatalysis, energy conversion, and storage and nanodevices [13–19]. Some investigations have indicated that noble metal Au nanoparticles deposited on TiO$_2$ surface can obviously enhance the photocatalytic performance via suppressing the recombination of electron-hole pairs, and also extending the light response in the visible region [14, 20, 21]. However, the practical applicability of such a composite structure is limited owing to exposing both the Au nanoparticles to reactants and the surrounding, while the corrosion or dissolution of the Au nanoparticles during the photocatalytic reaction is likely to limit the usage of Au, especially for long-term working [13]. To overcome this shortcoming, the Au nanoparticles embeded or enwrapped by the TiO$_2$ aggregates have attracted considerable attention, and significant advances have been made in recent years. The TiO$_2$ aggregates offer protection for Au nanoparticles against dissolution, photocorrosion, and chemical corrosion under extreme conditions, as well as being used for decomposition of dye wastewater. Among the various phases of TiO$_2$, anatase has proven to be the most suitable one due to its
two distinctive performances. One is that the photogenerated electrons of anatase can rapidly reduce Au ions at interface [22]. The other is that the conduction band of anatase can transfer the photogenerated electrons of the excited Au to adsorbed O2 and then to form active superoxide radical anions [23–25].

Several techniques, such as sol-gel method, chemical vapor deposition, laser vaporization, modified impregnation, precipitation-reduction, and photoreduction, have been developed to design and modulate the Au-TiO2 composite nanoparticles. The preferred and versatile method is the sol-gel process, which has many advantages, such as simple equipment required, flexible control of pore structures, and the concentration of Au [26–30]. Usually, the TiO2 aggregates obtained by this method are amorphous in nature and calcination temperatures higher than 400°C are required to realize the phase transformation from amorphous to anatase. However, such high calcination temperatures will not only lead to the increase of crystallite size and the decrease of specific surface areas and pore volume, but also induce the heat-aggregation of Au nanoparticles. Generally, the Au particles have exhibited highly catalytic activity only with smaller nanoscale size [31–34]. So, in order to obtain crystallized Au-TiO2 composite nanoparticles with higher specific surface areas and pore volume, the temperature of phase transformation from amorphous to anatase must be lowered.

In this paper, a novel and simple method, a low-temperature spray hydrolysis assisted with photoreduction technique, was proposed for the synthesis of Au-TiO2 nanocomposites. The Au nanoparticles (Au colloids) were prepared by photoreduction technique using a 350 W xenon lamp as a light source. Au-TiO2 nanocomposites were prepared by spraying the mixed solution of Ti(OC4H9)4 and EtOH into hot distilled water in the presence of Au nanoparticles (ca. 90°C). The formation of hydroxyl radicals (•OH) on the surface of visible-light illuminated Au-TiO2 composite samples was detected by the photoluminescence technique using terephthalic acid as a probe molecule. The photocatalytic activity of the Au-TiO2 composite samples was evaluated by photocatalytic decolorization of RhB aqueous solution under visible-light irradiation (λ > 420 nm).

2. Experimental

2.1. Preparation of Au Nanoparticles. All the involved chemicals in this study were purchased from Shanghai Chemical Reagent Co., Ltd, and used without further purification. Typically, 10 mL aqueous solution of sodium citrate (38.8 mM) was added into 100 mL aqueous solution of HAuCl4·4H2O (1 mM) under vigorous stirring. A 350 W xenon lamp was used a light source to irradiate the mixed solution during the reduction process. The color of the solution was gradually changed from light-yellow to fuchsia. After irradiation for another 30 mins, it was cooled to room temperature.

2.2. Preparation of Au-TiO2 Composite Nanoparticles. In a typical synthesis [35], 10 mL of mixed solution of Tetrabutyl titanate (TBOT) and ethanol (volume ratio of TBOT to ethanol, 1:4) was sprayed into a 250 mL beaker containing 200 mL hot distilled water (90°C), in which the added above Au nanoparticles was used as Au source. The corresponding nominal atomic ratio of Au to Ti, which hereafter was designated as R_{Au}, was 0.003, 0.006, 0.015, 0.030, and 0.060 nominal atomic % (at. %). After further stirring for 2 h at 90°C, the resultant mixed solution was aged for 2 h at 90°C. The aged wet precipitates were filtrated, rinsed with distilled water and absolute alcohol for two times. Then the precipitates were dried in a vacuum oven at 80°C for 10 h and finally ground to obtain Au-TiO2 composite samples. As a control experiment, the pristine TiO2 product was also prepared under the same condition but without adding Au nanoparticles.

2.3. Characterization. X-ray diffraction (XRD) patterns obtained on a D/Max-RB X-ray diffractometer (Rigaku, Japan) using Cu Kα irradiation at a scan rate (2θ) of 0.05° s−1 were used to determine the phase structure of the obtained samples. The average crystallite sizes of anatase were determined according to the Scherrer equation using the full width at half maximum (FWHM) data of anatase phase after correcting the instrumental broadening. Morphology observation was performed on a S-4800 field emission scanning electron microscope (FESEM, Hitachi, Japan). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) observation were conducted using a JEM 2100F microscope at an accelerating voltage of 200 kV. The Brunauer-Emmett-Teller surface areas (S_{BET}) of the samples were analyzed by nitrogen adsorption in a Micromeritics ASAP 2020 nitrogen adsorption apparatus (USA). All the samples were degassed at 80°C prior to nitrogen adsorption measurements. The BET surface area was determined by a multipoint BET method using the adsorption data in the relative pressure (P/P0) range of 0.05 to 0.30. Desorption isotherm was used to determine the pore size distribution via the Barret-Joyner-Halender (BJH) method, assuming a cylindrical pore modal [36]. The nitrogen adsorption volume at the relative pressure (P/P0) of 0.994 was used to determine the pore volume and average pore size. UV-visible absorbance spectra of Au-TiO2 composite nanoparticles were obtained for the dry-pressed disk samples with a UV-visible spectrophotometer (UV-2550, Shimadzu, Japan). BaSO4 was used as a absorption standard in the UV-visible absorbance experiment. X-ray photoelectron spectroscopy (XPS) measurements were performed on a Kratos XSAM800 XPS system with Cu Kα source. During the experiments the pressure in the chambers was about 10−7 Pa. All the binding energies were referenced to the C1s peak at 284.8 eV of the surface adventitious carbon.

2.4. Analysis of Hydroxyl Radical (•OH). The formation of hydroxyl radical (•OH) on the surface of photoirradiated Au-TiO2 is detected by photoluminescence (PL) technique using terephthalic acid as a probe molecule. Terephthalic acid readily reacts with •OH to produce highly fluorescent product, 2-hydroxyterephthalic acid [37, 38]. This technique has
been widely used in radiation chemistry, sonochemistry, and biochemistry [37–39] for the detection of •OH generated in water. The intensity of the PL peak of 2-hydroxyterephthalic acid is in proportional to the amount of •OH radicals produced in water [37–39]. The optimal concentration of terephthalic acid solution was about $5 \times 10^{-4}$ M in a diluted NaOH aqueous solution ($2 \times 10^{-3}$ M). This method relies on the PL signal at 425 nm of the hydroxylation of terephthalic acid with •OH generated at the water/TiO$_2$ interface. The method is rapid, sensitive, and specific, only needs a simple standard PL instrumentation.

In order to measure the concentration of hydroxyl radical (•OH) of the prepared samples, the typical experimental procedures are as follows: Au-TiO$_2$ composite samples were weighed and then uniformly dispersed into 20 mL distilled water under 10 min ultrasonic shaking to form TiO$_2$ suspension. The photocatalyst was prepared by coating the above aqueous suspension of TiO$_2$ samples onto one dish with a diameter of about 9.0 cm. The weight of catalysts used for each experiment was kept 0.1 g. The dishes containing composite powders were dried in an oven at 80°C for about 2 h to evaporate the water and then cooled to room temperature before being used. A 20 mL of the $5 \times 10^{-4}$ M terephthalic acid aqueous solution with a concentration of 2

$$2 \times 10^{-3} \text{ M NaOH was added into the coated dish, which was then irradiated with a 15 W daylight lamp. In order to ensure only the visible light irradiation, each dish was covered with an ultraviolet cutoff filter ($\lambda > 420 \text{ nm}$). PL spectra of generated 2-hydroxyterephthalic acid were measured on a fluorescence spectrophotometer (F-7000, Hitachi, Japan) using a 315 nm excitation wavelength at a scan speed of 1200 nm min$^{-1}$ with the PMT voltage of 700 V. The width of excitation slit and emission slit were 1.0 nm.}

2.5. Photocatalytic Activity. The visible-light photocatalytic activity of the Au-TiO$_2$ composite samples was evaluated by photocatalytic decolorization of RhB aqueous solution. The detailed procedure was similar to our previous works [40]. Here, the weight of the photocatalyst used was kept at about 0.1 g. A 15 W daylight lamp (6 cm above the dishes, just mentioned above) was used as a light source and each dish was covered with an ultraviolet cutoff filter. The absorbance of RhB was determined by a UV-visible spectrophotometer (UV2550, Shimadzu, Japan). The concentration of RhB ($c$) was in proportion to the light absorbance ($A$) at 550 nm according to Beer-Lambert law. For the Rhodamine-B aqueous with a concentration of $1 \times 10^{-5}$ M, its photocatalytic decolorization is a pseudofirst-order reaction and its kinetics may be expressed as [41, 42]:

$$\ln \frac{A_0}{A_t} = kt,$$

where $k$ is the apparent rate constant, and $A_0$ and $A_t$ are the initial and reaction absorbance of RhB aqueous, respectively.

3. Results and Discussion

3.1. Phase Structures. XRD was used to investigate the changes of phase structure of the as-prepared Au-TiO$_2$ nano-composites and pure TiO$_2$. Figure 1 shows the effects of $R_{\text{Au}}$ on phase structures of the Au-TiO$_2$ composite powders. It can be seen that the diffraction peaks of all samples were indexed with the anatase phase of TiO$_2$, which was consistent with our previous work [35]. With increasing the $R_{\text{Au}}$, the intensities of anatase peaks steadily become weaker and the width of the diffraction peaks ($2\theta = 25.3^\circ$) of anatase slightly became wider. This was probably due to the fact that Au nanoparticles suppress the crystallization of TiO$_2$ xerogel powders. However, the XRD diffraction peaks of Au could not be seen in the Figure 1, which was attributed to the fact that the content of Au was so low that it could not be detected by XRD diffractometer.

The microstructure of the Au-TiO$_2$ composite powder was further studied by TEM and HRTEM. Figure 2(a) shows the TEM image of the $R_{\text{Au}}$-0.060 composite sample. It can be observed from Figure 2(a) that the nanocrystallite appears as an agglomerated status, and mesoporous structures without a long-range order. The size of the primary particles estimated from the TEM image was about $6 \pm 1$ nm, which was in accord with the value of crystallize size (6.0 nm) calculated from XRD pattern using the Scherrer equation (as shown in Table 1). Figure 2(b) shows the corresponding HRTEM image of the $R_{\text{Au}}$-0.060 composite powder. Further observation shows that all the particles are crystallized as evidence from the well-resolved Au (111) (0.25 nm) and TiO$_2$ (101) (0.35 nm) crystalline lattices as shown in the inset of Figure 2(b) [42–44].

Effects of the $R_{\text{Au}}$ on the surface morphology of the prepared Au-TiO$_2$ composite powders were characterized by FESEM. Figure 3 illustrates FESEM images of all the Au-TiO$_2$ composite powders, indicating their nonuniform growth and particle size distribution in spherical morphology, which changed slightly with the different Au modification. This agglomeration process was attributed to Van der Waals
Figure 2: TEM (a) and HRTEM (b) images of the $R_{Au}$-0.060 composite powder.

Figure 3: FESEM images of the Au-TiO$_2$ composite powders prepared with different $R_{Au}$: (a) 0, (b) 0.003, (c) 0.015, and (d) 0.060.
forces. In order to reduce the surface energy, the primary particles have a tendency to form an agglomerate, by forming nearly spherical or equiaxed agglomerates, in a minimum surface-to-volume ratio and hence minimum surface free energy can be achieved. All the Au-TiO2 composite powders are composed of such nanoscale particles, indicating that the composite powders prepared by this low-temperature spray method possess large specific surface area and the high volume fraction of atoms located both on the surface and at the grain boundaries. Therefore, this Au-TiO2 composite nanocrystalline can provide more active sites for catalysis. Furthermore, all the Au-TiO2 composite powders have roughness surface accompanied with many mesopores. In general, such nanoscale particles with such diameter will be more promising because light harvesting can be further enhanced. In this respect, nanocrystalline Au-TiO2 composite particles with this special morphology can enhance the absorbance of the light resulting in the enhancement of photocatalytic activity. Further observations indicated that the powders without Au nanoparticles modification (pristine TiO2) have large particles. With increasing the RAu, the average size of aggregated particles decreases.

3.2. UV-Visible Absorbance Spectra. Figure 4 shows UV-Vis absorbance spectra for the composite Au-TiO2 samples. A significant increase at wavelengths longer than 400 nm could be attributed to the plasmon resonance absorbance of the Au nanoparticles [22, 44]. The Au-TiO2 composite powders compared with the pristine TiO2 powders have a slight red shift in the band gap transition. Moreover, a characteristic absorbance signal at 530 nm give the valid evidence that the Au phase existed in the products, though direct observation by XRD failed [24, 33, 45–47]. Further observation indicates that the intensity of the surface plasmon absorbance increase with increasing RAu, which is probably due to the more Au nanoparticles embed in TiO2 aggregates. The enhanced light absorption in the visible light region can therefore increase the quantities of photogenerated electrons and holes that participate in photocatalytic reactions. In order to verify the chemical state of Au element in the Au-TiO2 composite powders, the RAu=0.060 sample was also characterized with XPS. High-resolution XPS spectrum (inset in Figure 4) shows binding energy of Au 4f7/2 (84.0 eV) and Au 4f5/2 at 87.7 eV, which are significantly different from Au+ (84.6 eV) and Au+ (87.0 eV). The result suggests that the Au species is in the metallic state [25, 44, 48, 49].

3.3. BET Specific Surface Areas and Pore Structure. All the prepared Au-TiO2 composite powders have similar nitrogen adsorption-desorption isotherms and pore size distribution curves. Therefore, Figure 5 only presents the nitrogen adsorption-desorption isotherm and pore size distribution (inset) of the RAu=0.015 composite powder.

Table 1 shows the effects of RAu on the physical properties of the Au-TiO2 composite powders. It can be seen that all the prepared samples possess large specific surface area (SBET > 200 m2/g) and big pore volume (VP > 0.30 cm3/g). With increasing RAu, the average pore sizes decrease from 8.1 to 5.4 nm while the specific surface area and pore volume change slightly. Generally, the heterogeneous photocatalysis is a surface-based process, and therefore a large surface area has positive effects on such a process. A larger surface...
area provides more surface active sites for the adsorption of reactant molecules, which thus make the photocatalytic process more efficient [50–53]. Moreover, the porous structure is believed to facilitate the transporting of reactant molecules and products through the interior space due to the interconnected porous networks, and it favor the harvesting of exciting light due to enlarged surface area and multiple scattering within the porous framework [54–56].

3.4. Hydroxyl Radical Analysis. The PL emission spectrum excited at 315 nm of terephthalic acid solution was measured every 30 min under visible-light irradiation. Figure 6(a) shows the changes of PL spectra of terephthalic acid solution with irradiation time. It can be seen that a gradual increase in PL intensity at wavelength range of 360–540 nm is observed with increasing irradiation time. Moreover, the generated spectra have the identical shape and peak position (at 425 nm). However, no PL increase is observed in the absence of visible-light or TiO2 samples. This suggests that the signal of PL is only caused by the reaction of terephthalic acid with •OH formed on the interface of the Au-TiO2/water during visible-light irradiation [37, 38].

Generally, the greater the formation rate of •OH radicals is, the higher separation efficiency of electron-hole pairs is achieved. So, the photocatalytic activity is in positive correlation to the formation rate of •OH radicals, namely, a faster formation rate of •OH radicals leads to a higher photocatalytic activity [38]. Figure 6(b) shows the dependence of PL intensity against irradiation time. It can be easily seen that at a fixed time (120 min), the formation rate of •OH radicals on the Au-TiO2 composite powders is larger than that of the pristine TiO2. This implies that the Au-TiO2 composite powders have higher photocatalytic activity than the pristine TiO2. Further observation shows that the order of the formation rate of •OH radicals formed on the surface of as-prepared samples is as follows: R_{Au-0.015} > R_{Au-0.006} > R_{Au-0.003} > R_{Au-0.030} > R_{Au-0.060} > R_{Au-0} (pristine TiO2), which suggests that the R_{Au} influences the formation rate of •OH radicals and there is an optimum ratio of Au to TiO2.

3.5. Photocatalytic Activity of the Au-TiO2 Composite Powders. The photocatalytic activity of the Au-TiO2 composite powders was evaluated by photocatalytic decolorization of Rhodamine-B aqueous solution under visible-light irradiation (λ > 420 nm) at room temperature. Figure 7 shows the relationship between the apparent rate constants (k) and R_{Au}. It can be seen that the R_{Au} has a great effect on the photocatalytic activity of the composite samples. Without
Table 1: Effects of RAu on physical properties of the Au-TiO₂ composite powders.

<table>
<thead>
<tr>
<th>Sample (RAu)</th>
<th>Phase content</th>
<th>Crystalline size/nm</th>
<th>BET surface area/m²/g</th>
<th>VM/cm³/g</th>
<th>Porosity (%)</th>
<th>Average pore size/nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Anatase</td>
<td>6.7</td>
<td>235.5</td>
<td>0.57</td>
<td>69.0</td>
<td>8.1</td>
</tr>
<tr>
<td>0.003</td>
<td>Anatase</td>
<td>6.7</td>
<td>208.4</td>
<td>0.46</td>
<td>64.2</td>
<td>7.5</td>
</tr>
<tr>
<td>0.006</td>
<td>Anatase</td>
<td>6.5</td>
<td>211.7</td>
<td>0.34</td>
<td>57.0</td>
<td>7.1</td>
</tr>
<tr>
<td>0.015</td>
<td>Anatase</td>
<td>6.5</td>
<td>205.0</td>
<td>0.43</td>
<td>62.6</td>
<td>5.7</td>
</tr>
<tr>
<td>0.030</td>
<td>Anatase</td>
<td>6.3</td>
<td>215.9</td>
<td>0.38</td>
<td>59.7</td>
<td>5.8</td>
</tr>
<tr>
<td>0.060</td>
<td>Anatase</td>
<td>6.0</td>
<td>212.4</td>
<td>0.34</td>
<td>57.0</td>
<td>5.4</td>
</tr>
</tbody>
</table>

"a:" Average crystalline size of TiO₂ was determined on the basis of the broadening of the anatase {101} diffraction peak using the Scherrer equation.
"b:" The BET surface area was determined by multipoint BET method using the adsorption data in the relative pressure (P/P₀) range of 0.05–0.2.
"c:" Pore volume, porosity, and average pore size were determined by nitrogen adsorption volume at the relative pressure of 0.994.

Au modification, the prepared pristine TiO₂ powders have the poor visible-light photocatalytic activity presumably due to the self-sensitization of RhB molecules, which extends the absorption of titania into the visible-light region [57]. Importantly, the photocatalytic activity of all Au-TiO₂ samples is higher than that of pristine TiO₂. Further observation shows the reaction rate increases with the increasing of Au content when the value of RAu is below 0.015, and the RAu ~ 0.015 sample has the best photocatalytic activity among these samples. Therefore, the appropriate modification of the gold nanoparticles can enhance the photocatalytic activity of TiO₂ remarkably, which is ascribed to the synergetic effect of the dye-photosensitized and the surface plasmon resonance of Au nanoparticles in the Au-TiO₂ nanocomposites.

The possible photocatalytic enhanced mechanism of the Au-TiO₂ nanocomposite powders can be explained according to the recently reported mechanism [4, 15, 22, 47]. The noble metal nanoparticles can be photoexcited by the visible light due to their plasmon resonance. Under visible-light irradiation, the electrons and holes can be formed on the surface of the Au nanoparticles, and then they are immediately separated via the following processes. The electrons transfer to the adsorbed oxygen molecules via the conduction band of TiO₂ and then are trapped through formation of superoxide radical anions. The superoxide radicals and the trapped electrons can combine to produce H₂O₂, finally forming hydroxyl radicals. Both hydroxyl radical and superoxide radical anions are strong oxidants which can oxidize the organic molecules (RhB) on the surface of the composite powders, resulting in the formation of intermediate organic species and subsequently complete oxidation of these species to water and carbon dioxide. These improvements would be beneficial to the photooxidation of RhB using the Au modified TiO₂ under visible-light irradiation. When the RAu reaches 0.030, the Au-TiO₂ composite powders show relatively lower photocatalytic activity, which is probably due to the fact that more Au nanoparticles may act as the centers of electron-hole recombination and reduce quantum efficiency [44].

Figure 8 shows the change of absorption spectra of RhB aqueous solution during its visible-light photocatalytic decolorization using the RAu-0.015 sample as the photocatalyst. It can be seen that the absorption peak at λ = 550 nm drops gradually with increasing visible-light irradiation time. After visible-light irradiation for ca. 180 minutes, the absorption peak of RhB aqueous solution is very weak and the color of the RhB aqueous solution changes from orange to near no color, indicating that the Au-TiO₂ composite powders can completely decolorize RhB aqueous solution under visible-light irradiation. Usually, the complete decolorization of RhB requires a long time due to the fact that its decolorization goes through two different stages: the ring cleavage in the initial photocatalytic degradation stage and subsequent oxidation of the fragments in the latter stage [40, 58]. Therefore, these composite powders prepared by this method could be useful for environmental applications such as air purification, water disinfection and purification, and hazardous waste remediation due to their cheap preparation process, controllable structure, large specific surface area, and high visible-light photocatalytic activity.

4. Conclusions

Au-TiO₂ nanocomposites can be facilely prepared by a simple spray hydrolytic method and assisted with photoreduction technique at 90°C. The light absorption, the
formation rates of hydroxyl radicals, and photocatalytic decolorization of RhB aqueous solution were significantly enhanced by those embedded Au nanoparticles in the Au-TiO₂ nanocomposites due to surface plasmon resonance of Au nanoparticles. The composite Rₐ₋=0.015 sample exhibited the best visible-light photocatalytic activity for photocatalytic decolorization of RhB aqueous solution due to the synergistic effects of the absorption shift into visible and improved efficiency of interfacial charge transfer process.

Acknowledgments

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