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# Photoreduction of carbon dioxide on quantized semiconductor nanoparticles in solution

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## Abstract

The results published so far show that semiconductor nanoparticles (Q-Sc) possess high activities as photocatalysts for reduction of $CO_2$ as compared with the corresponding bulk semiconductor photocatalysts. Several factors which influence photoreduction behaviors of carbon dioxide on size-quantized semiconductor nanoparticles (Q-Sc) in solution systems are described, which include the size of the Q-Sc, the charged condition of stabilizers used for Q-Sc, and the polarity of solvents used in photoreduction studies of $CO_2$. © 1997 Elsevier Science B.V.

*Keywords:* Semiconductor nanoparticles; Photocatalysts; $CO_2$ reduction

## 1. Introduction

Photoinduced reduction of $CO_2$ on semiconductor photocatalysts in solution systems, which are usually coupled with oxidation of water, are among the most desired reactions of chemical conversion and storage of solar energy into chemical energy. Attempts to achieve this were made in the 1980s using bulk semiconductor particles of high stability in aqueous solutions such as $TiO_2$, SrTiO3, and SiC. However, efficient reduction of $CO_2$ was not achieved in those cases. Table 1 gives several results on photoreduction of $CO_2$ using bulk semiconductor photocatalysts. It can be seen that more than two kinds of reduction products were obtained in many cases. Although not shown in the table, the amount of products obtained were usually too small to allow reliable analysis results and for this reason, it is not always easy to reproduce the reported results.

The use of sacrificial electron donors which have high activities for donating electrons to irradiated semiconductor photocatalysts, such as trimethylamine and triethanolamine, slightly improved the situation. It allows the use of semiconductor particles of low electrochemical stability such as ZnS and CdS as the photocatalysts besides the semiconductors of high electrochemical stability. The sacrificial electron donors can pick up quickly photogenerated positive holes from the semiconductor photocatalysts and can prevent the photodissolution of the photocatalysts. Furthermore, the photoreduction rate of $CO_2$ seems to have been enhanced by the use of the sacrificial electron donors, suggesting that if no sacrificial electron donors are used, oxidation of water by photogenerated positive holes determines the rate of $CO_2$ photoreduction on bulk semiconductor photocatalysts. Unfortunately, however, few articles have been published on photoreduction of $CO_2$ using bulk semiconductor photocatalysts in the presence of sacrificial electron donors.

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Table 1
Photoreduction of $CO_2$ using bulk semiconductor photocatalysts in aqueous solutions

| Semiconductor/Loaded catalyst | Sacrificial electron donor | Products | Reference |
|---|---|---|---|
| SiC, GaP, $TiO_2$, etc. | | HCHO, $CH_3OH$ | [1] |
| $SrTiO_3$, $WO_3$, $TiO_2$, etc. | | HCHO, $CH_3OH$, $CH_4$ | [2] |
| $SrTiO_3$/ Oxides of Rh, Pt, Ir | | $HCOO^-$, (HCHO, $CH_3OH$, $CH_3CHO$, $C_2H_5OH$)[a] | [3] |
| $BaTiO_3$, $LiNbO_3$ | | $CH_3CHO$, HCOOH, HCHO | [4] |
| $TiO_2/RuO_2$ | | HCOOH, $CH_3OH$, HCHO | [5] |
| $TiO_2$/Cu | | $CH_4$, $C_2H_6$ | [6] |
| $TiO_2/RuO_2$/Ru[b] | | $CH_4$ | [7] |
| $TiO_2$/Rh | | HCOOH, HCHO, $CH_3OH$ | [8] |
| $TiO_2$/Pd | | HCOOH | [9] |
| $TiO_2$/Pt | | HCHO | [10] |
| $TiO_2$, $SrTiO_3$/Pt, Rh, Ru, Ag | | CO, $CH_4$, HCOOH | [11] |
| $TiO_2$/Suspended Cu[c] | (Supercritical $CO_2$) | HCOOH, $CH_3OH$ | [12] |
| $CaFe_2O_4$ | | $CH_3OH$, HCHO | [13] |
| $Cu_2O_xH_2O$ | | $CH_3OH$, HCHO | [14] |
| ZnS | 2,5-dihydrofran | HCOOH | [15] |
| CdS | TEACl[d] | Glyoxylic acid, acetic acid,<br>HCOOH, $CH_3OH$, $CH_3CHO$ | [16] |
| CdS | EDTA,TEOA[e] | HCOOH, HCHO | [17] |

[a] Products given in the parenthesis are minor products.
[b] The experiments were carried out in gaseous atmosphere containing $H_2$ and $CO_2$.
[c] Copper powder was suspended in solution together with $TiO_2$.
[d] Tetraethylammonium chloride.
[e] Triethanol amine.

Recently photochemistry of Q-Sc is emerging, and it was revealed that Q-Sc has in general higher activities as photocatalysts than the corresponding bulk semiconductors for several heterogeneous reactions including photoreduction of $CO_2$. In the following, recent results on $CO_2$ photoreduction using Q-Sc photocatalysts is described.

## 2. Effects of the size of Q-Sc on the rate of photoreduction of $CO_2$

The first report on the use of Q-Sc as a photocatalyst for reduction of $CO_2$ was published in 1984 by the Henglein group [18]. They reported that $CO_2$ was photoreduced to formate on Q-ZnS in the presence of 2-propanol with the quantum efficiency of 80%, which had never been reported by that time for photoinduced reactions on semiconductor photocatalysts. Reexamination of the photocatalytic reduction of $CO_2$ under almost the same experimental conditions as those reported by the Henglein group revealed that Q-ZnS has certainly high activities for $CO_2$ photoreduction to formate [19], but the quantum efficiency obtained for the formate production was not 80% but 23%. Almost the same quantum efficiency was reported by Yanagida and coworkers who used Q-ZnS as the photocatalyst in the presence of $H_2PO_4^-$ and $SH^-$ [20]. Interestingly, it was found that the quantum efficiency for the formate production was influenced by the bandgap of Q-ZnS. Q-ZnS having different bandgap values were prepared by changing the molar ratio of $Zn(ClO_4)_2$ to $Na_2S$ used in the preparation of Q-ZnS, and it was found that the greater the molar ratio, the smaller the particles or the greater the bandgap. The bandgap ($E_g$) was determined by applying the absorbance of the spectra of ZnS colloids in its onset region to Eq. (1), and the particle size of the Q-ZnS was determined by applying the obtained bandgap to Eq. (2), which was derived by Brus [21]. So far several theoretical approaches have been published concerning the relations between the particle sizes of Q-Sc and the bandgap values [21–24].

$$\alpha h\nu = A(h\nu - E_g)^{1/2} \tag{1}$$

$$E_g = E + 2\hbar^2\pi^2/d^2(1/m_e^* + 1/m_h^*) - 3.6e^2/\epsilon d, \tag{2}$$

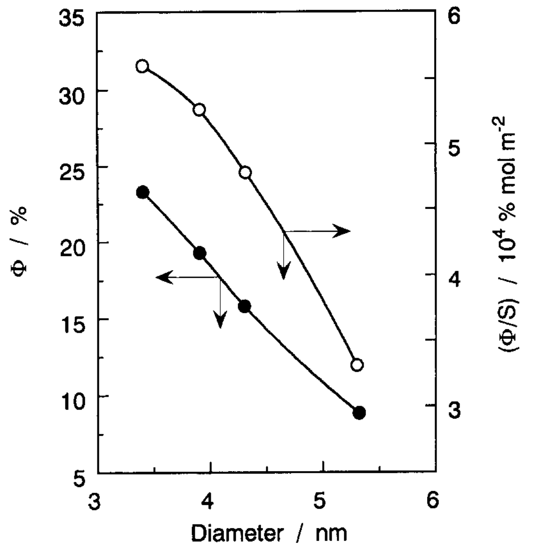

Fig. 1. The quantum efficiency ($\Phi$) for formate production and the efficiency divided by the total surface area of the photocatalyst ($\Phi/S$) as a function of the size of the photocatalyst. Irradiation of $CO_2$-saturated aqueous solution (pH 7) containing 1 mol $dm^{-3}$ 2-propanol and $4\times10^{-4}$ mol $dm^{-3}$ Q-ZnS with a 500 W high pressure mercury arc lamp for 60 min.

where $\alpha$ is the absorbance, $\nu$ is the wave number, $m_e$ and $m_h$ are the effective mass of an electron and a hole, $\epsilon$ and $E$ the dielectric constant of ZnS and the bulk bandgap energy, respectively. Fig. 1 shows the quantum efficiency obtained for the formate production from $CO_2$ as a function of the particle size of the Q-ZnS photocatalyst. According to the results shown in this figure, the quantum efficiency became high with decreasing the particle size of the photocatalyst.

The following factors may be conceivable as the principal causes for the particle size dependence of the quantum efficiency observed here: (1) Since the bandgap becomes large with decreasing the size of the particle, both the oxidizing power and reducing power of the photocatalyst becomes great with decreasing the size of the photocatalyst. (2) With decreasing the particle size, the total surface area of the photocatalysts per weight unit increases, and (3) the affinity of adsorbing $CO_2$ onto photocatalyst surfaces seems to become high with decreasing particle size, as judged from the finding that the tendency of agglomeration of Q-Sc becomes larger with decreasing the particle size. The factor which is the most likely among these three is then a matter for discussion. Let the first factor be discussed first. Fig. 2 shows the energy diagram of the bulk ZnS [25] relative to several reduction reactions of $CO_2$ in aqueous solutions at pH 7. It is seen that the bottom of the conduction band of ZnS is more than 1 V more negative than the reduction reaction of $CO_2$ to formate. This potential difference is regarded as the driving force of the reduction reaction, and is said to be large enough to induce most of electrochemical reduction reactions. Accordingly, it is said that the bulk ZnS has a high reduction power. Since the bandgap of Q-Sc which showed the largest blue shifts in the onset of the absorption spectra was 4.1 eV at most, while that of the bulk ZnS is 3.8 eV, we do not expect to have a large contribution of the size quantization to the increase in the potential difference between the conduction band of Q-Sc and the reduction reaction of $CO_2$ to formate. Furthermore, the bulk ZnS photocatalyst prepared with the same technique used for the preparation of Q-ZnS but without using $SiO_2$ as the stabilizer did not show any remarkable activities for reduction of $CO_2$. Considering these, the first factor does not play an important role in the apparent increase of photocatalytic activities with decreasing the particle size. In order to get information on the contribution of the second factor, the quantum efficiency was divided by the surface area of the particles, and the obtained values are plotted as a function of the particle size of photocatalyst. The results are also included in Fig. 2. It is evident that

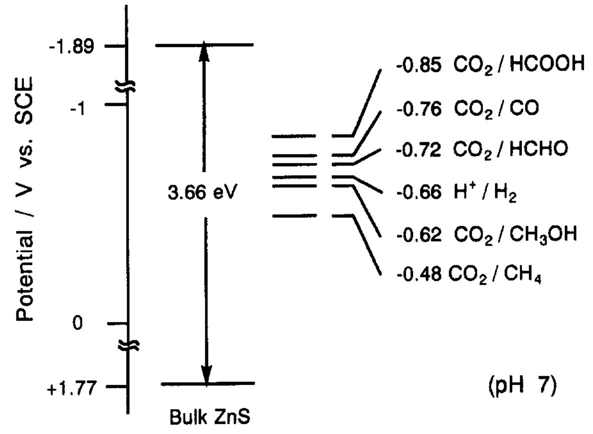

Fig. 2. Energetic correlation of bulk ZnS and reduction reactions of $CO_2$ to several kinds of products in aqueous solutions at pH 7.

the increase in the photocatalytic activities of Q-ZnS with decreasing the particle size was more than the increase in the surface area [19], suggesting that the increase of the surface area alone cannot explain well the apparent increase in the photocatalytic activities observed. Accordingly it is concluded that the third factor is involved in the apparent enhancement of the photocatalytic activities with decreasing the particle size.

It is well established in a variety of photocatalytic reactions using bulk semiconductor photocatalysts that loading of electrocatalysts onto the semiconductor photocatalyst surfaces is useful as a means for enhancing the activities of photocatalysts. The same was found to be true for Q-Sc photocatalysts. The loading of Cd in 0.025 mol% onto Q-ZnS with use of the photodeposition technique resulted in the enhancement of the quantum efficiency for reduction of $CO_2$ to formate from 16% to 32% [27].

The increase in the apparent photocatalytic activities with decreasing the particle size of the photocatalyst was also observed at Q-$TiO_2$ prepared in silica matrix using the sol–gel technique, which is denoted here as Q-$TiO_2/SiO_2$ [26]. It was found that the Q-$TiO_2/SiO_2$ having the greater bandgap or the smaller particle size was prepared by using the smaller molar ratio of $Ti(OEt)_4$ to $Si(OEt)_4$ in the preparation of the sol. As shown in Table 2, $CO_2$ was mostly reduced to formate at the Q-$TiO_2/SiO_2$ in an aqueous solution containing 1 mol $dm^{-3}$ 2-propanol as a sacrificial electron donor.

Table 2
Quantum efficiency at 280 nm for production of reduction products of $CO_2$ at Q-$TiO_2/SiO_2$ in the presence of 2-propanol

| Ti/Si ratio | $E_g$ (eV) | Quantum efficiency (%) | | |
|---|---|---|---|---|
| | | $HCOO^-$ | $CH_4$ | $C_2H_4$ |
| 0.10 | 4.0 | 9.0 | 0.50 | 0.1 |
| 0.048 | 4.1 | 12.7 | 1.5 | 1.8 |
| 0.0027 | 4.33 | 14.2 | 3.4 | 2.3 |

## 3. Effect of charged condition of stabilizers for Q-Sc on reduction behaviors of $CO_2$

Q-Sc was prepared by mixing the same volume of $6\times10^{-4}$ mol $dm^{-3}$ $Cd(ClO_4)_2$ and $4\times10^{-4}$ mol $dm^{-3}$ in the presence of various kinds of polymers as the stabilizing agents. The amount of the polymers used was such that as to give the concentration of $8\times10^{-4}$ mol $dm^{-3}$ in the monomer unit. The bandgap of the prepared Q-CdS was determined by applying the absorption spectra to the relation given by Eq. (1). The results obtained in the photoreduction of $CO_2$ are shown in Table 3 [28], which suggests that the stabilizing agents having negative charges favor the photoreduction of $CO_2$ to formate, while the CO production occurs more easily with use of positively charged stabilizers. Also suggested from comparison of the irradiation time shown in Table 3 for the CO production and for the formate production with each other is that the rate of formate production is higher than that

Table 3
Results of photoreduction of $CO_2$ using Q-CdS in the presence of a variety of polymers as the stabilizing agents[a]

| Stabilizer | Quantity of charge[b] (mol $dm^{-3}$) | $E_g$[c] (eV) | Irradiation time (h) | Products (μmol) | | |
|---|---|---|---|---|---|---|
| | | | | $HCOO^-$ | CO | $H_2$ |
| Poly(sodium vinylsulfonate) | Negative $7.8\times10^{-4}$ | 2.68 | 4 | 0.73 | 0 | 0.090 |
| Poly(sodium 4-styrenesulfonate) | Negative $8\times10^{-4}$ | 2.66 | 4 | 0.88 | 0 | 0.046 |
| Polyacrylic acid | Negative $2.4\times10^{-4}$ | 2.80 | 3 | 1.77 | 0 | 0.59 |
| Poly(allylamine hydrochloride) | Positive $8\times10^{-4}$ | 2.80 | 16 | 0 | 0.75 | 0 |
| Polybrene | Positive $8\times10^{-4}$ | 2.71 | 9 | 0 | 0.11 | 0 |
| Polyethyleneimine | Positive $4\times10^{-4}$ | 2.85 | 6 | 0 | 0.40 | 0 |

[a] Experiments were carried out using 4 $cm^3$ of $4\times10^{-4}$ M Q-CdS in the presence of $8\times10^{-3}$ M stabilizer in the monomer unit, 1 M 2-propanol, $2\times10^{-4}$ M $Cd^{2+}$ and saturated amount of $CO_2$. Solution pH was 5.5. Irradiation was carried out using a 500 W xenon lamp as a light source at 0.51 W $cm^{-2}$ and lights of the wavelengths shorter than 310 nm was cut off.

[b] The quantity of charges of the polymers in the photoreduction experiments of $CO_2$.

[c] The bandgap value of Q-CdS prepared in the presence of the stabilizer.

of CO. Apparently the charged conditions of the stabilizers influence the reduction behaviors of $CO_2$. One plausible explanation of the role of the charged condition of the stabilizers in the photoreduction of $CO_2$ may be like this. The reduction of $CO_2$ occurs with adsorption on the photocatalyst surfaces. When a reduction intermediate such as $CO_2^{\cdot}$ is produced, its adsorption on the photocatalyst surfaces will be influenced by the charged conditions of the stabilizers. If there is a negatively charged stabilizer on the Q-CdS surfaces, the adsorption of $CO_2^{\cdot}$ onto the photocatalyst surface may occur through the carbon atom, while if a positively charged stabilizer is present, both carbon and an oxygen of $CO_2^{\cdot}$ may be involved in its adsorption.

## 4. Effect of polarity of solvents used in photoreduction of $CO_2$

The solvents used in photoreduction studies of $CO_2$ seem to have some effect on the reduction behaviors of $CO_2$, as shown in Fig. 3 for the use of the Q-$TiO_2$/$SiO_2$ as the photocatalyst. The ordinate of the figure gives the fraction of formate as a reduction product to the sum of the products which consisted of formate and CO. The experiments were carried out using 1 mol dm$^{-3}$ 2-propanol as the sacrificial electron donor and no water was intentionally added when nonaqueous solvents were used. It is important to describe here that almost the same dependency of the fraction of formate on the dielectric constant of the solvents as that shown in Fig. 3 was obtained for the commercially available P-25 $TiO_2$, except for the use of propylene carbonate which gave the methanol production at P-25 $TiO_2$ [29]. If it is assumed that the solvent used plays a crucial role in the photoreduction of $CO_2$, the relative ratio of the reduction products must have the same dependency on the dielectric constant of the solvents that are shown in Fig. 3 even if other kinds of semiconductors are used as the photocatalysts. So far several papers have been published on the photoreduction of $CO_2$ in aqueous solutions and formate was mostly obtained as the major reduction products at Q-ZnS [18,26,27], Q-CdS [27], and SiC [30] as the photocatalysts.

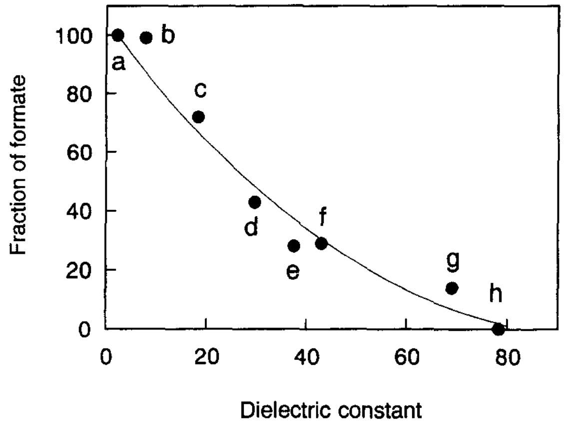

Fig. 3. Influence of the dielectric constant of solvents used in photoreduction studies of $CO_2$ on the fraction of formate of the total reduction products which consisted of formate and CO. The experiments were carried out by irradiating a Q-$TiO_2$-immobilized silica film of 2 cm$^2$ in 5 ml of various kinds of solutions containing 1 M 2-propanol as the sacrificial electron donor. The photocatalyst film was prepared by using the sol–gel technique, and 7 μmol $TiO_2$ was contained in 70 μmol $SiO_2$ film. Solvents used: a: Carbon tetrachloride, b: Dichloromethane, c: 2-Propanol, d: Ethylene glycol monoethyl ether, e: Acetonitrile, f: Sulfolane, g: Propylene carbonate, and h: Water.

## 5. Photoreduction of $CO_2$ in propylene carbonate solutions

When P-25 $TiO_2$ was used as a photocatalyst for reducing $CO_2$ in propylene carbonate solution in the presence of 2-propanol as the sacrificial electron donor, methanol was produced as the reduction product. The methanol production at much higher rates was obtained using Q-$TiO_2$ immobilized in polyvinylpyrrolidinone gel film as the photocatalyst [31]. The photocatalyst used in that case was prepared by hydrolyzing 1 ml portion of titanium tetraisopropoxide in 50 ml of 2-propanol containing 0.6 ml of water, 0.04 ml of 1 mol dm$^{-3}$ HCl and 0.5 wt% of polyvinylpyrrolidinone which worked as the stabilizing agent, followed by applying electrophoresis using a platinum mesh cathode [32]. The photoreduction experiments of $CO_2$ were carried out both by immersing the entire surfaces of the photocatalyst in the solution and by contacting one face of the photocatalyst film with $CO_2$ and the other face with the solution. In both cases acetone was obtained as the sole oxidation product, while $Ti^{3+}$, $H_2$ and methanol

Table 4
Photoinduced reduction of $CO_2$ on semiconductor nanoparticle photocatalysts

| Q-Sc/catalyst/(stabilizer) | Sacrificial electron donor/Solvent | Reduction products | Remarks | Reference |
|---|---|---|---|---|
| $ZnS/(SiO_2)$ | 2-Propanol/water | HCOOH | $QE^a$=80% | [18] |
| $ZnS/(SiO_2)$ | 2-Propanol/water | HCOOH | QE=23%, | [19] |
| ZnS | $H_2PO_4^-$, $SH^-$/water | HCOOH | QE=23% | [20] |
| CdS | $TEA^b$/DMSO | CO, $H_2$ | QE=9.8% | [33] |
| CdS | TEA, $BP^c$/DMF | Benzyl acid | | [34] |
| $TiO_2$/(Vycor glass)$^d$ | Water vapor | $CH_4$, $CH_3OH$ | | [35] |
| ZnS/Cd/(HMP)$^e$ | 2-Propanol/water | HCOOH | QE=42% | [27] |
| CdS/In$^f$ | TEA/DMF | HCOOH, CO | | [36] |
| $TiO_2/(SiO_2)^d$ | 2-Propanol/water | HCOOH<br>$CH_4$, $C_2H_6$ | | [26] |
| CdS/polymer | 2-Propanol/ water | HCOOH><br>CO | For polymers of negative charge<br>For polymers of positive charge | [29] |
| $TiO_2$/(PVPD)$^g$ | 2-Propanol | $CH_3OH$ | | [31] |

[a] Quantum efficiency (%).

[b] Triethylamine.

[c] Benzophenone.

[d] Immobilization in solid support.

[e] Hexamethaphosphate.

[f] The photoreduction experiments was carried out in the presence of $In^{3+}$ in the solution.

[g] Polyvinylpyrrolidinone gel film.

were obtained as the reduction products. The chemical stoichiometry of the photoinduced reaction seems good, since the number of holes involved in the production of acetone was equal to that involved in the reduction reactions. Since higher production rates of these were obtained when the photocatalyst film was contacted with both the solution and gaseous $CO_2$, the amount of $CO_2$ around the Q-$TiO_2$ photocatalysts seems to determine the overall rate of the photoinduced reaction. The methanol production from reduction of $CO_2$ was confirmed by labeling experiments using $^{13}CO_2$. Presently it is not known why the selective photoreduction of $CO_2$ to methanol occurs in the propylene carbonate solution.

## 6. Conclusion

Results obtained by using Q-Sc as the photocatalysts are collected in Table 4 which shows that the sacrificial electron donors were used in all cases. Since Q-Sc has high activities for photoreduction of $CO_2$, a fairly high amount of reduction products that allowed reliable chemical analyses was obtained. From the viewpoint of $CO_2$ fixation, however, the use of sacrificial electron donors is not recommended, and water must be used as the hole scavenger. Unfortunately we have no good technique to induce oxidation of water efficiently on semiconductor photocatalysts. Water photolysis on the semiconductor photocatalysts have not yet been successful due to difficulty in evolving oxygen from the photocatalyst surfaces. If this problem is solved, then the photoreduction of $CO_2$ using semiconductor photocatalysts will be easily achieved without using any sacrificial electron donors. In this sense, a breakthrough that allows the achievement of water photolysis is desired, and this subject is still most important for studies on semiconductor photocatalysis.

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