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PHOTOCATALYTIC OXIDATION OF HYDROGEN SULFIDE ABSORBED IN AQUEOUS SOLUTIONS

CdS powder was used to study the photocatalytic oxidation of hydrogen sulfide absorbed in aqueous solution. H_2S was oxidized to polysulfides with a simultaneous evolution of H_2 on platinized CdS suspensions.

The production of H_2 depended on the following parameters: initial concentration of H_2S in the solution, pH of the suspension, radiant flux, and reaction temperature. The yield of H_2 increased with increase of the platinum deposit and reached a maximum activity at 2%-wt of platinum. Twenty-hour irradiation yielded an approximately 90% conversion of H_2S . The turnover numbers (in H_2 yield with regard to CdS) of 4.7 and 25 have been determined in the S^{2-} and HS^- suspensions, respectively. The activation energy for the photooxidation of H_2S approached 8.5 kJ/mol. The reaction mechanisms are also discussed.

NOTATION

- d_i — grain diameter, nm,
- d_s — surface weighed mean diameter, nm,
- e^- — electron,
- e_{tr}^- — trapped electron,
- CB — conduction band,
- h — hole,
- h_{tr}^+ — trapped hole,
- N.H.E. — hydrogen reference electrode,
- N_i — number of grains of diameter d_i ,
- t_I — illumination time,
- VB — valence band.

SUBSCRIPTS

- n — number of ions,
- tr — trap.

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1. INTRODUCTION

In recent years, considerable interest has been focussed on the development of systems converting solar energy into a useful form. Photoelectrochemical approaches to solar energy conversion are based on band-gap excitation of photoactive semiconductors in contact with liquids. Photoelectrochemical systems can be configured either to produce electricity or to drive important reactions, i.e., photoelectrosynthesis and photodecomposition.

Significant progress has been achieved due to conducting homogeneous photoredox reactions with semiconductor suspensions or colloids. Photoelectrochemical systems have been used to carry out reactions, which may be of utility in wastewater treatment. The literature contains references to the application of particulate semiconductors, e.g., TiO_2 , $\text{TiO}_2/\text{RuO}_2$, to photocatalytic oxidation of cyanides [1], trichloroethylene [2], 4-chlorophenol [3] or other substances (just to mention a few). But the photocatalysts reported are able to absorb only ultraviolet light, which limits their use to solar applications. To eliminate this drawback, it is necessary to develop photocatalytic systems based on smaller band-gap semiconductors, as CdS.

Recently, CdS colloids and suspensions have received considerable attention because of their photocatalytic activity under visible light irradiation. Notable achievements in this area have been, for example, water cleavage on $\text{CdS}/\text{RuO}_2/\text{Pt}$ [4], production of H_2 using sulfide solutions [5], as well as decomposition of aldehydes and alcohols [6].

Removal of gaseous pollutants (e.g., H_2S) by absorption in solutions raises disposal problems. The effluent is usually a dilute solution which cannot be discharged to the recipient stream without further treatment. Conventional methods require a concentration step. This study aimed at developing such a method that would enable conversion of the persisting pollutants to a useful form. In the method proposed in this paper, H_2S is converted to H_2 and polysulfides. Of these, H_2 is a clean fuel and polysulfides can be easily disposed of. The reaction is endergonic and involves light energy as driving force.

2. EXPERIMENTAL

2.1. MATERIALS AND APPARATUS

All chemicals used in this study were analytical-grade reagents. Doubly-distilled water was used to prepare all solutions and suspensions.

All experiments were performed in two water-jacketed, Simax cylindrical photoreactors (25 mm \times 2 mm, I.D.). The top of these reactors was equipped with a

gas tight silicon septum and a side arm that permitted passing argon gas both through and over the solution. The volume of the suspension was 25 cm³.

Steady-state irradiations were carried out using a LH-21 halogen lamp (Polam). The incident light flux was measured using L-02 (Sanopan) and LM-2 (Carl Zeiss) radiometers.

The gas phase hydrogen concentration was measured by a gas chromatograph (Elwro), with a catharometric detector and a stainless steel column (2 m × 4 mm) packed with Porapak Q (0.149–0.125 mm), at 295 K. The carrier gas was argon, with a flow rate of 20 cm³/min.

2.2. PROCEDURE

CdS particles (of luminophore grade, Poch) were surface platinized by photo-reduction of hexachloroplatinic acid (Mennica Państwowa). A 100 mg portion of CdS was suspended in ~ 20 cm³ of redistilled water. The suspension was treated with 0.05 to 5 cm³ of 0.01 mol/dm³ hexachloroplatinic acid and with 0.91 cm³ of 0.5 mol/dm³ HCHO solution in 2:3 V/V methanol–water mixture. The mixture was then diluted to 25 cm³ with redistilled water.

At a constant temperature of 298 ± 0.5 K, the suspension was flushed with pure argon (20 dm³/h) during 15 min. for oxygen removal prior to irradiation. The mixture was then stirred with a magnetic stirrer and irradiated for 2 h at 130 mJ/cm²s.

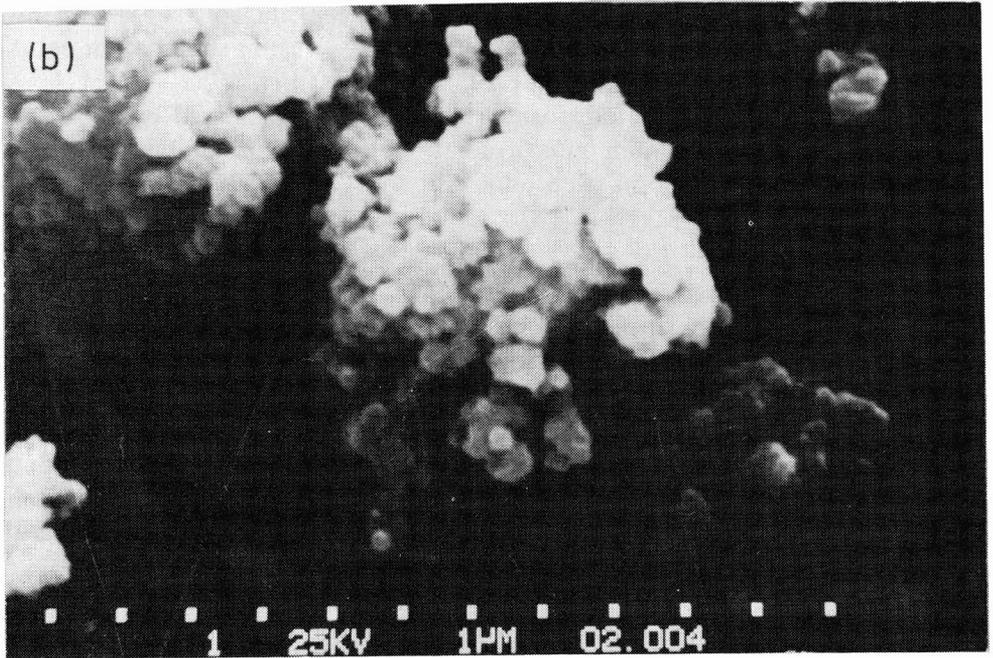
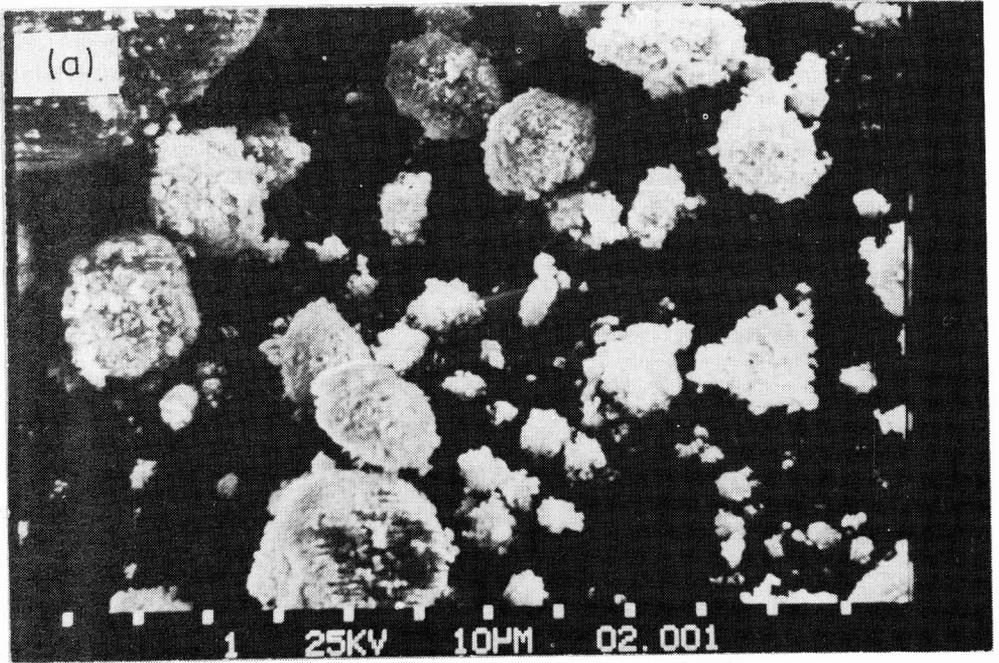
After turning off the light, the suspension was centrifuged at 4000 r.p.m. for 15 min. The residual concentration of H₂PtCl₆ · 6H₂O in the supernatant solution was measured colorimetrically (Spekol 20, Carl Zeiss) [7].

The actual amount of photodeposited platinum was calculated by subtracting the residual amount of platinum (after irradiation) from the original net platinum in the solution before irradiation. Reproducibility was below 7%.

Absorption of hydrogen sulfide

H₂S was produced by dropwise addition of 20% H₂SO₄ on to Na₂S crystals (Reachim). Three absorbing solutions were used [8], [9]: 10% NaOH solution, 50% NaOH solution and redistilled water. Water pH was adjusted by addition of 10% NaOH solution to the value between 9 and 10. Prior to H₂S exposure the samples were deaerated by argon bubbling for 45 min at a flow rate of approximately 10 cm³/min. Argon bubbling was continued during H₂S exposure. The amount of H₂S produced was washed by bubbling through a saturated solution of Ba(OH)₂ before being transferred by argon to the absorbing solution.

The amount of H₂S in the solutions was determined by iodometric titration [8] in which a starch was used as an indicator.



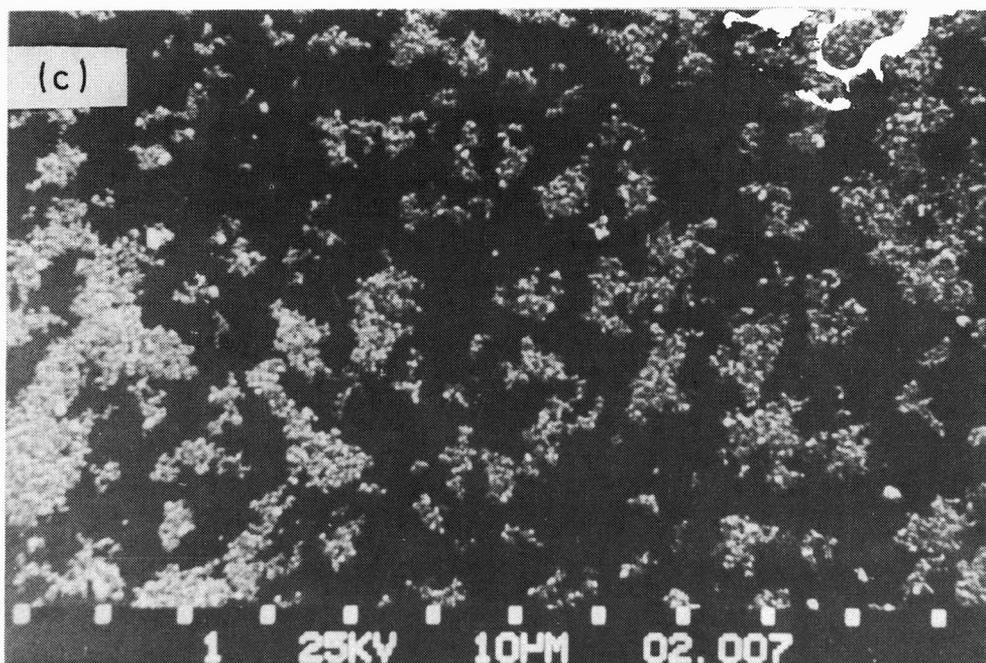


Fig. 1 a-c. Scanning electron micrographs of CdS particles

a, b - CdS powder (as supplied), c - sonicated CdS particles suspended in water (sonication time 2h)

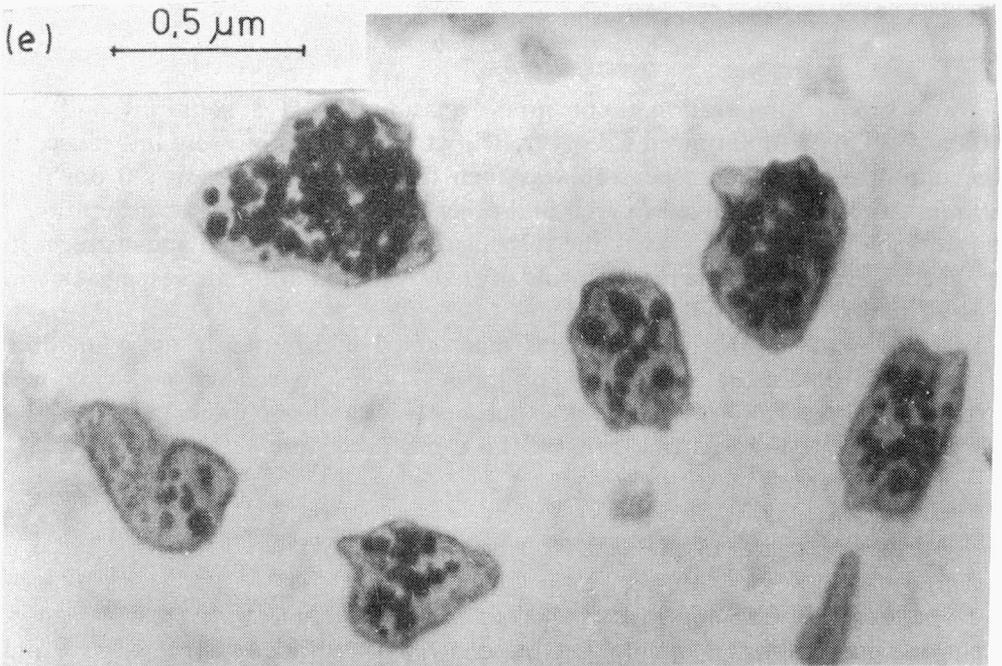
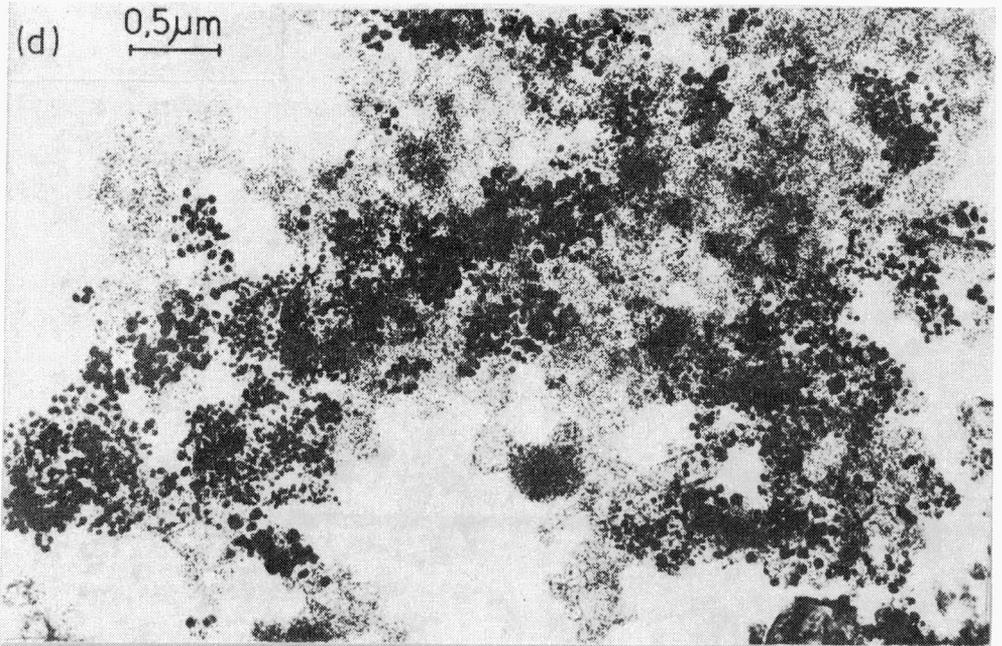
Photooxidation of hydrogen sulfide

For sample preparation, appropriate amounts of H_2S solutions and about 100 mg of CdS/Pt (platinized CdS, 1 to 4%-wt Pt) suspension were introduced into the photoreactor. The suspension was then deaerated with argon ($30 \text{ dm}^3/\text{h}$) for 30 min to remove dissolved oxygen and other absorbed gases. After deaeration, the photoreactor was tightly closed, the suspension was thermostated and stirred with a magnetic stirring bar. The reaction was started by illuminating the suspension with a LH-21 lamp.

The amount of H_2 produced was determined intermittently as a function of illumination time using a gas chromatograph. The results were reproducible within experimental error of $\pm 10\%$. Blank experiments without a photocatalyst and in the absence of H_2S were carried out by the same procedure.

3. RESULTS AND DISCUSSION

The texture of CdS and Pt, as well as their particle diameters, were obtained from electron micrographs taken with a Stereoscan 180 (Cambridge Instruments) scanning



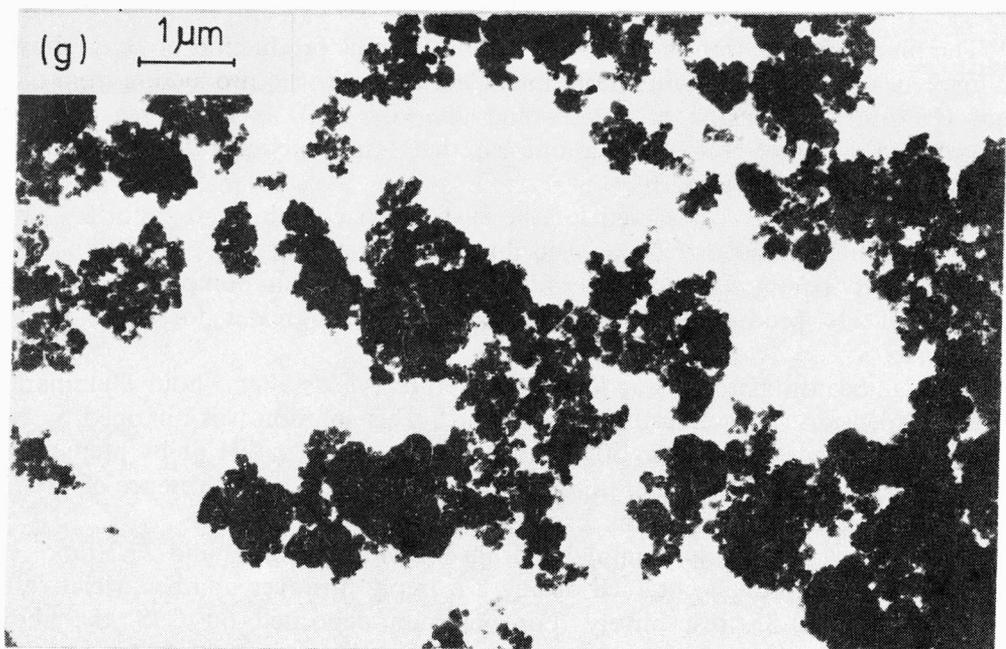
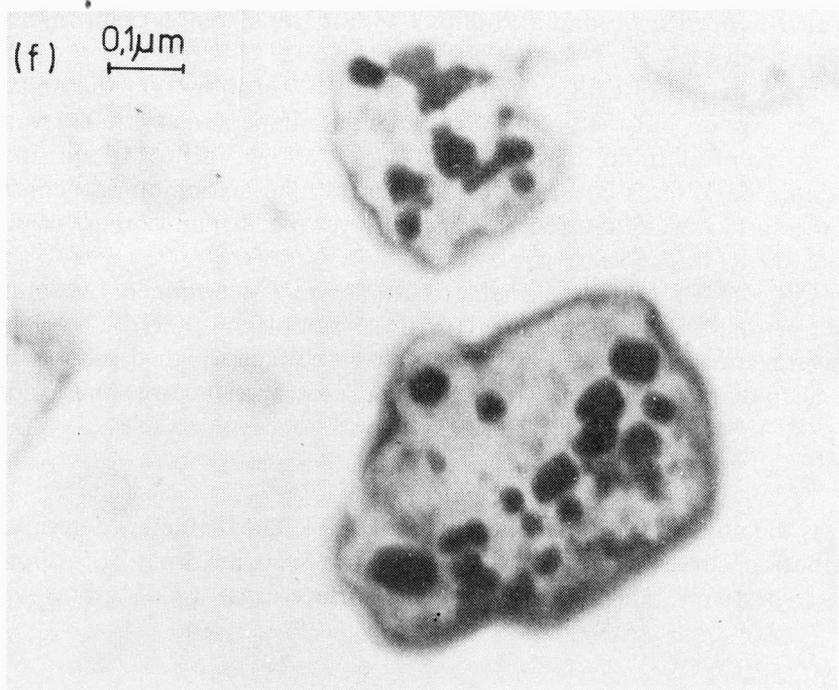


Fig. 1 d-g. Transmission electron micrographs of CdS particles

d, e, f - platinized CdS/Pt (4% Pt), g - CdS/Pt (10% Pt).

Platinum aggregates (dark spots) were photodeposited on CdS by photoreduction of $\text{H}_2\text{PtCl}_6/\text{HCHO}$ aqueous solution. Deposition time 2 h, temperature 298 K, flux 130 $\text{mJ}/\text{cm}^2\text{s}$

electron microscope (SEM) and a Phillips EM-301 transmission electron microscope (TE), respectively.

SEM micrographs of CdS particles (figs. 1a, 1b, 1c) show that the uniformity of the particles is poor, but the grains have a regular shape and size. Observation by a TE microscope of platinum loaded CdS (4% Pt), shown in figs. 1d, 1e, 1f, revealed that platinum dark spots covered the CdS powder surface inhomogeneously. Some parts of the surface were covered with a thin layer (fig. 1d) and others were covered with a relatively thick layer of platinum.

The CdS and Pt particle size distributions were determined by counting the particles (on micrographs) contained in three samples.

The average diameters were determined from surface weighed plots and surface weighed mean diameters, d_s (eq. 1), assuming that the particles are spherical in shape. Hence,

$$d_s = \frac{\sum N_i d_i^3}{\sum N_i d_i^2} \quad (1)$$

where N_i is the number of grains of diameter d_i [11]. The diameters determined from the distribution curves averaged ~ 400 nm and ~ 30 nm for CdS grains and Pt particles, respectively. The estimated specific surface area of CdS grains was about $3.3 \text{ m}^2/\text{g}$.

3.1. PRODUCTION OF HYDROGEN

The photo-degradation of H_2S with a simultaneous production of H_2 is shown in fig. 2 as a function of illumination time. Curve 1 shows H_2 production from a $0.1 \text{ mol HS}^-/\text{dm}^3$ suspension at pH 11 and $298 \pm 0.5 \text{ K}$. The initial reaction rate approached $6.4 \mu\text{mol H}_2/\text{h}$. After about 4 h, the H_2 production rate decreased and became steady at $2.4 \mu\text{mol/h}$.

A similar trend was observed for the suspension containing $0.1 \text{ mol S}^{2-}/\text{dm}^3$ (curve 2). After a rapid rise in the yield during the first hour, the production of H_2 became steady and increased linearly with prolonged illumination periods. Both the amount of H_2 produced and the reaction rate are greater for a suspension containing S^{2-} .

No significant difference was found between the yields after 5-hour illumination of the suspension containing $0.1 \text{ mol S}^{2-}/\text{dm}^3$. This solution was obtained by two methods: by absorbing H_2S in 50% aqueous solution of NaOH or by preparation from commercial Na_2S . Neither in the absence of H_2S nor in the absence of CdS/Pt was hydrogen detected in blank experiments.

The total yields of $146.7 \mu\text{mol H}_2$ (from $0.1 \text{ mol S}^{2-}/\text{dm}^3$) and $27.7 \mu\text{mol H}_2$ (from $0.1 \text{ mol HS}^-/\text{dm}^3$) obtained during 8 h imply turnover numbers (relative to CdS) of 4.7 and 25, respectively. For platinum deposited on CdS, the above mentioned hydrogen yields imply turnover numbers of $6.99 \cdot 10^{-2}$ and 2.7, respectively.

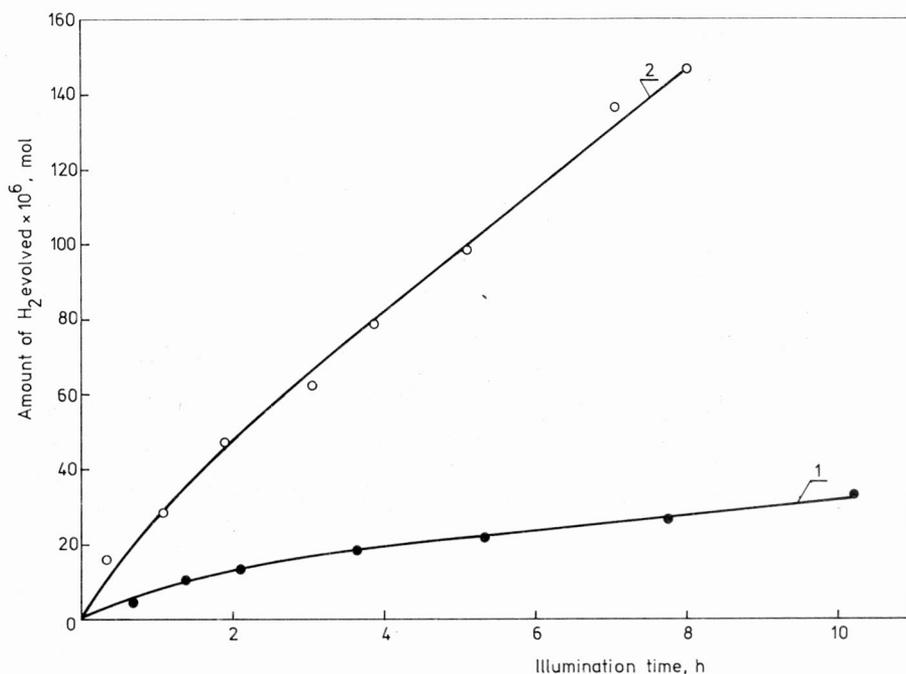


Fig. 2. Hydrogen evolution as a function of illumination time

pH 11, temperature 298 K, flux 130 mJ/cm² s

1 - 0.1 mol HS⁻/dm³, 100 mg CdS/Pt (2% Pt), 2 - 0.1 mol S²⁻/dm³, 100 mg CdS/Pt (4% Pt)

3.2. EFFECT OF PLATINUM LOADING

The relationship between the Pt deposit on the surface of CdS particles and the yield of H₂ production is given in fig. 3. As shown by this plot, H₂ yield increased with the increasing deposit to reach a maximum activity at 2%-wt Pt. Further deposition of Pt led to a decrease in H₂ yield. The data in fig. 3 indicate how the performance of the system improves with the increasing activation of CdS particles by Pt particles. This is attributed to the fact that H₂ evolution on CdS requires an over-voltage higher by about 300 mV than does evolution on Pt [16]. Hence, when Pt is in contact with CdS, it acts as a cathode at which H₂ is easily evolved.

In addition, deposition of Pt also leads to the modification of the electronic properties of CdS resulting from the ohmic contact. In consequence, this induces bending of both conduction and valence bands at CdS surfaces, which brings about an efficient separation of hole and electron pairs [10].

The progressive regression of activity observed above 2% Pt is probably due to the fact that the platinum fraction becomes sufficiently large, i.e., 10% (fig. 1g), to act

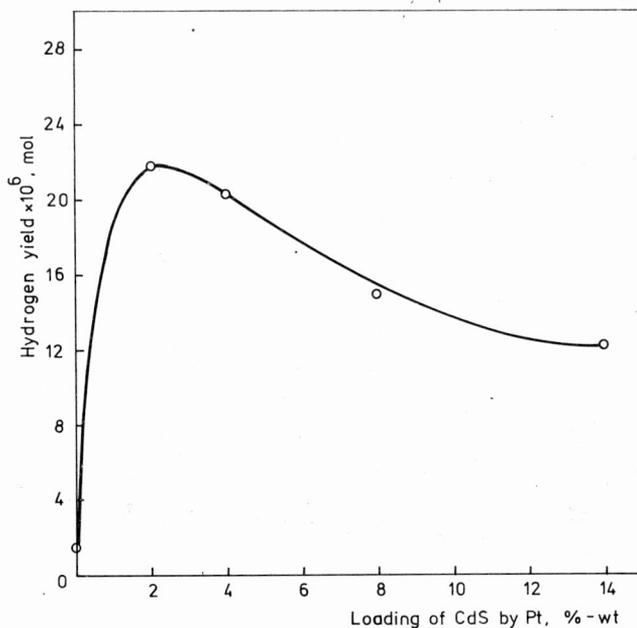


Fig. 3. Effect of Pt loading of CdS on the H₂ yield
0.1 mol HS⁻/dm³, 298 K, illumination time $t_i = 300$ min, flux 130 mJ/cm²s

as an optical filter for the active radiation and/or detrimental charge recombination at the platinum particles. These two factors obviously increase with the increasing metal content for a constant particle size [11].

3.3. EFFECT OF INITIAL CONCENTRATION

Figure 4 relates the yield of H₂ formation to the initial concentration of HS⁻. A sharp increase in the H₂ yield can be observed in the concentration range from 0 to 10 mmol HS⁻/dm³. The rate of H₂ generation decreases when initial concentration increases from 10 to 50 mmol/dm³. Once this value has been achieved, further increase in initial concentration is without any significant effect.

Figure 5 shows the H₂ production as a function of illumination time at various concentrations of S²⁻. After 3 h of irradiation, the rate of H₂ production was almost constant. In the concentration range from 0.01 to 0.25 mol S²⁻/dm³ a gradual increase in rate was observed. The mean rate of H₂ formation, measured after 100 min of irradiation, varied from 0.7 μmol·H₂/h at 1·10⁻³ mol S²⁻/dm³ to 38 μmol H₂/h at 0.2 mol S²⁻/dm³.

The data in figs. 4 and 5 may be partly explained by the fact that, owing to the extremely fast electron-hole recombination in CdS, only adsorbed molecules or ions

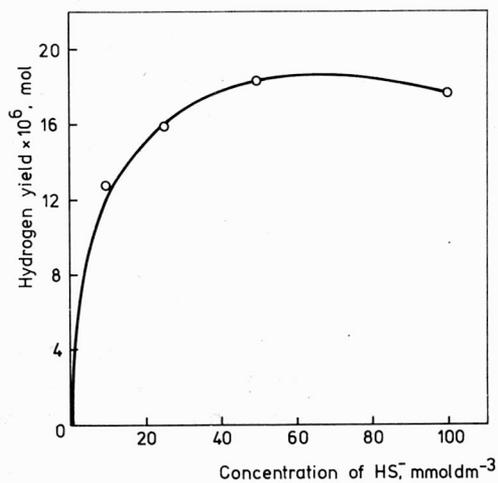


Fig. 4. Dependence of H_2 yield on the initial HS^- concentration
CdS/Pt (2% Pt), 298 K, $t_i = 200$ min

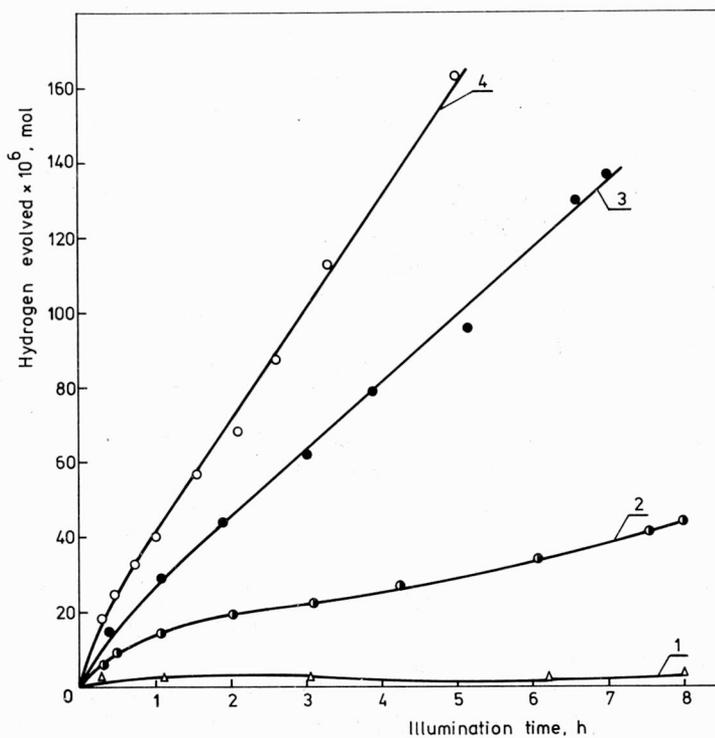


Fig. 5. Influence of initial S^{2-} concentration on the H_2 yield

1 - 0.001 mol $\text{S}^{2-}/\text{dm}^3$, 2 - 0.01 mol $\text{S}^{2-}/\text{dm}^3$, 3 - 0.1 mol $\text{S}^{2-}/\text{dm}^3$, 4 - 0.25 mol $\text{S}^{2-}/\text{dm}^3$, CdS/Pt (4% Pt)

can react with photogenerated charges. If the saturation limit is not reached, the amount of adsorbed species on the photocatalyst increases with increasing HS^- and S^{2-} concentration. Consuming the photogenerated holes, these anions enhance charge separation which is one of the rate determining processes.

Additionally, S^{2-} is known to prevent or minimize oxidation of lattice S^{2-} ions in CdS [12].

3.4. CONSUMPTION OF HS^-

Figure 6 illustrates the change in HS^- concentration and the concomitant H_2 yield as a function of illumination time. A fast consumption of HS^- was observed in the first 3 h. Later HS^- concentration continued to decrease with longer illumination

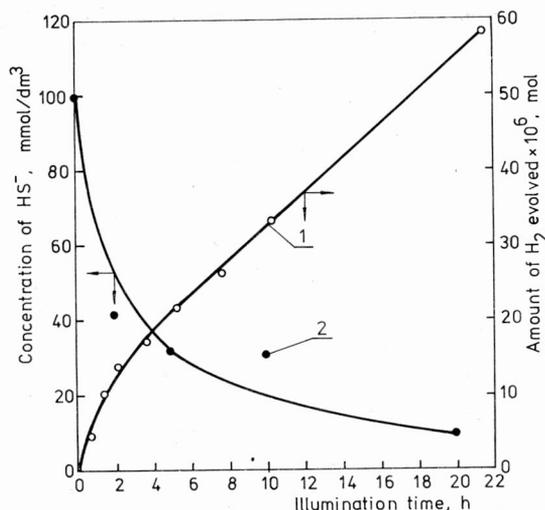


Fig. 6. Hydrogen production (1) and the simultaneous HS^- consumption (2) as a function of illumination time

All other conditions as in fig. 3

times. After 20 h a conversion of about 90% was noticed. The shapes of the curves indicate that the reaction rate was high in the first 3 h to drop with longer illumination periods. It is seen in fig. 6 that the amount of H_2S consumed can be correlated with the amount of H_2 evolved. At this stage, no attempt was made to determine the amount of polysulfides in the reacting mixtures, because they undergo a back reaction [20] according to eq. (11) (see further in the text).

3.5. EFFECT OF RADIANT FLUX ON HYDROGEN PRODUCTION

Figure 7 shows that H_2 evolution is proportional to the intensity of the light applied. The initial rate of H_2 evolution increased from 0.03 to $0.7 \mu\text{mol} \cdot H_2/\text{min}$ upon increasing the light intensity from 50 to $190 \text{ mJ}/\text{cm}^2\text{s}$. This variation indicates that saturation of the catalyst by photons has not been reached. The results may be

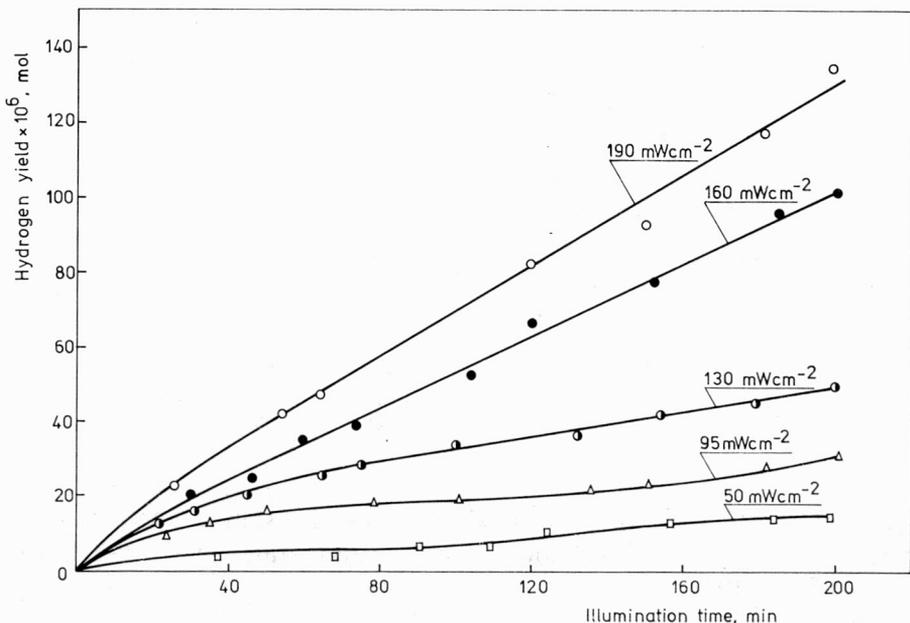


Fig. 7. Effect of radiant flux on H_2 generation

$0.25 \text{ mol } S^{2-}/\text{dm}^3$, 298 K , CdS/Pt (4% Pt)

rationalized in terms of band-gap excitation of CdS particles. Assuming that H_2 is formed in monophotonic reactions, i.e., that one electron is involved in H^+ reduction (excluding multiphotonic processes), the rate at which electron-hole pairs are formed is proportional to the incident flux of efficient photons.

3.6. pH-DEPENDENCE

The pH-dependence of H_2 formation is presented in fig. 8. NaOH solution and $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ buffer solution were used to adjust the pH of the suspensions. The H_2 yield increased from 20.3 to $41.7 \mu\text{mol}$ upon increasing pH from 10.8 to 13.1 . This finding can be explained in terms of negative shift of CdS flat band potential with

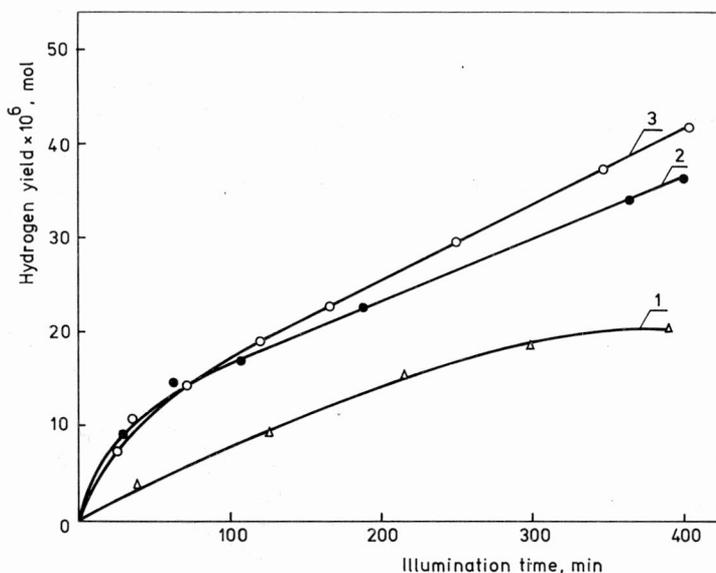


Fig. 8. Influence of pH on H_2 production

$0.01 S^{2-}$, 298 K, $130 \text{ mJ/cm}^2\text{s}$

1 - pH 10.8, 2 - pH 11.7, 3 - pH 13.1, CdS/Pt (4% Pt)

increasing pH. CdS is known [13] to exhibit a negative shift of about -40 mV per pH unit. This negative shift enhances charge transfer rates at the liquid semiconductor interface [19].

3.7. TEMPERATURE EFFECT ON THE HYDROGEN YIELD

Figure 9 gives the temperature dependence of H_2 yield. As shown by this curve, the rate of H_2 production increases when temperature rises from 292 to 228 K (by virtue of the Arrhenius plot). The overall experimental activation energy, calculated from the slope of the tangent of this plot, was 8.5 kJ/mol .

The influence of temperature was studied only for the suspension containing S^{2-} , since the second dissociation constant for H_2S shows a poor temperature-dependence [15].

The influence of temperature on the H_2 yield can be partly explained by the fact that all temperature-dependent reaction steps (i.e., adsorption, desorption, surface migrations and rearrangement), which are at the same time rate limiting, improve with increasing temperature. For example, the over-voltage of H_2 on platinum decreases by $0.02\text{--}0.03 \text{ V}$ per each 10 deg rise [16].

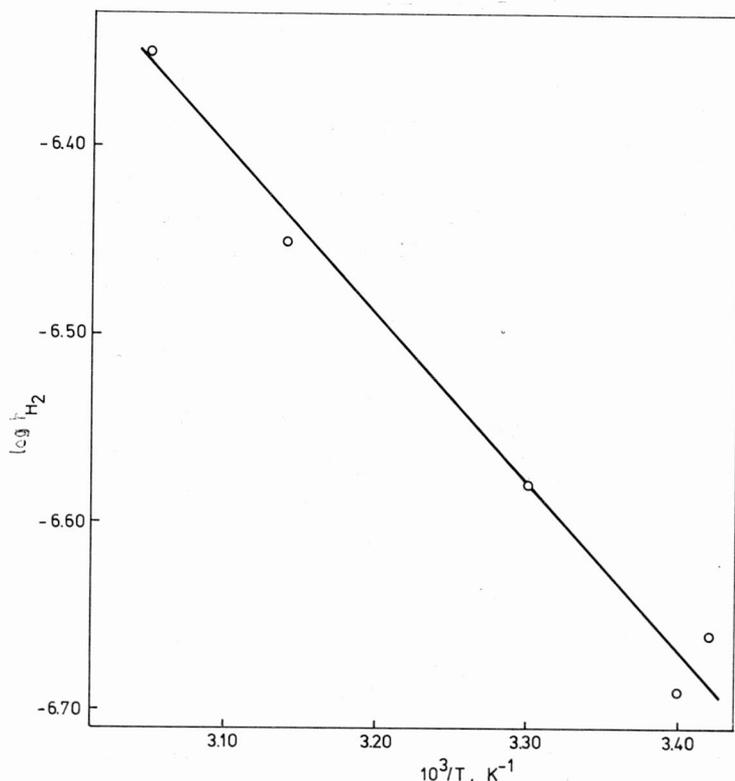


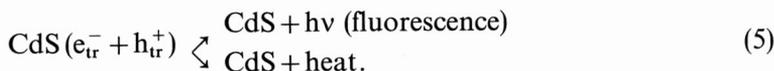
Fig. 9. Effect of temperature on H_2 yield
 $0.25 \text{ mol } S^{2-}/\text{dm}^3$, pH 12.5, $130 \text{ mJ}/\text{cm}^2\text{s}$, CdS/Pt (4% Pt)

3.8. REACTION MECHANISM

CdS is an *n*-type semiconductor characterized by a band-gap of 2.4 eV and a position of the valence and conduction band edge around +1.5 and -0.9 V (vs. NHE, at pH 7), respectively. Flat band potential E_{FB} at this pH approaches -0.66 V [17].

Irradiation of CdS particles with a light of an energy equal to or greater than the band gap energy accounts for the absorption of light by these particles, as well as for the creation of holes (h^+) in the valence band (VB) and for the generation of electrons (e^-) in the conduction band (CB), (eq. 2). Charge separation is followed by their trapping in shallow traps (tr) at the powder particle-solution interface (eqs. 3 and 4, fig. 10)





As it can be seen from eq. (5), there are three combinations of processes: 1) most of the trapped carriers recombine, 2) the electrons can be scavenged by reducible species (e.g., H^+ or O_2 in the solution), and 3) the holes can be scavenged by oxidizable species (e.g., HS^- , S^{2-} or H_2O).

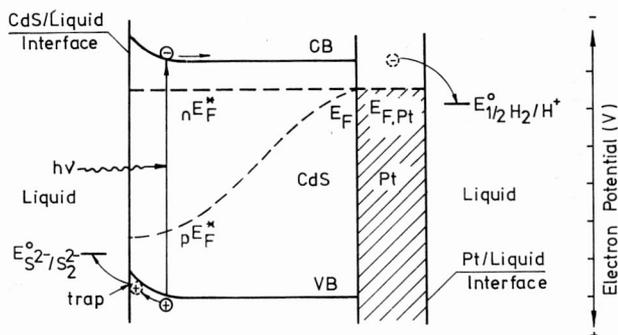
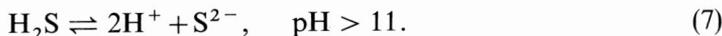
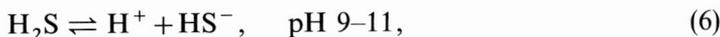


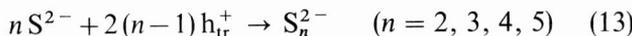
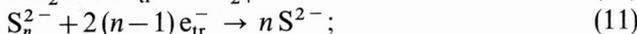
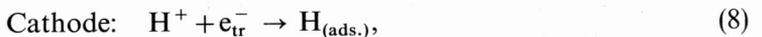
Fig. 10. Schematic energy-level diagram of CdS/Pt under illumination

CB - conduction band, VB - valence band, E_F - Fermi level; ${}_nE_F^*$ - electron Fermi level, ${}_pE_F^*$ - hole Fermi level

Taking these all into account, the mechanism governing the production of H_2 and the oxidation of H_2S can be explained as follows: H_2S dissociates in aqueous alkaline solutions according to eqs. (6) and (7)



The CdS flat band potential is sufficient to reduce protons and water in the presence of platinum as a catalyst (eq. (8)–(9))



where $\text{H}_{(\text{ads.})}$ denotes adsorbed hydrogen on the catalyst surface.

Since its oxidation potential is higher than that of HS^- or S^{2-} [18] the oxidation of water is energetically unfavourable. Hence, it is the oxidation of HS^- or S^{2-} that should be regarded as a thermodynamically favourable process. These two processes run according to reactions (12) and (13), respectively.

The species are oxidized to sulfur and polysulfides. Polysulfides may compete for electrons according to reaction (11). With longer irradiation times, this competition may influence the course of the reaction. The overall reaction corresponds to the oxidation of H_2S with photoproduction of H_2 by quanta of visible light and CdS/Pt catalyst.

4. CONCLUSION

The basic conclusions from this study may be summarized as follows:

1. H_2S absorbed in aqueous solutions was oxidized to polysulfides with a simultaneous evolution of H_2 on platinized CdS suspensions. The amount of H_2S consumed was correlated with the amount of H_2 evolved. Twenty-hour irradiation yielded an approximately 90% conversion of H_2S . The activation energy for the process was about 8.5 kJ/mol. The yield of H_2 reached optimum efficiency at 2%-wt Pt deposit on CdS .

2. The photocatalytic reactions investigated may give a promising method to remove H_2S from industrial wastewater streams with simultaneous generation of clean fuel, i.e., H_2 .

3. The semiconductor photocatalytic systems provide a promising way to convert solar energy into useful form, i.e., storable chemical energy. Since redox reactions are possible to run in such systems, they have potential viability in wastewater treatment with a simultaneous use of waste materials as reactants.

ACKNOWLEDGEMENTS

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FOTOKATALITYCZNE UTLENIANIE SIARKOWODORU ZAABSORBOWANEGO W ROZTWORACH WODNYCH

Badano fotokatalityczne utlenianie H_2S zaabsorbowanego w roztworach wodnych, stosując proszek CdS jako fotokatalizator. W wyniku utleniania siarkowodoru powstały wielosiarczki przy równoczesnym wydzielaniu wodoru na platynizowanych zawiesinach CdS. Ilość wydzielonego wodoru zależała od początkowego stężenia H_2S w roztworach, pH zawiesiny CdS, natężenia światła, temperatury oraz zawartości osadzonej platyny.

Naświetlając przez 20 godzin zawiesinę CdS 2% Pt strumieniem światła o natężeniu $1,3 \text{ kJ/m}^2 \cdot \text{s}$ otrzymano 90% utlenienie siarkowodoru do wielosiarczków. Energia aktywacji tej reakcji wynosiła około $8,5 \text{ kJ/mol}$. Przedyskutowano mechanizm reakcji utleniania siarkowodoru z równoczesnym wydzielaniem molekularnego wodoru.

ФОТОКАТАЛИТИЧЕСКОЕ ОКИСЛЕНИЕ СЕРОВОДОРОДА, АБСОРБИРУЕМОГО В ВОДНЫХ РАСТВОРАХ

Исследовано фотокаталитическое окисление сероводорода, абсорбируемого в водных растворах при применении порошка CdS в качестве фотокатализатора. В результате окисления сероводорода образованы полисульфиды при одновременном выделении водорода на платинированных суспензиях CdS. Количество выделенного водорода зависело от начальной концентрации сероводорода в растворах, pH суспензии CdS, силы света, температуры, а также от содержания осаждённой платины.

Освещая в течение 20 часов суспензию Cds 2% Pt световым потоком силы света $1,3 \text{ kJ/m}^2 \cdot \text{s}$, получили 90% окисление сероводорода до полисульфидов. Энергия активации этой реакции составляла $8,5 \text{ kJ/mol}$. Обсуждён механизм реакции окисления сероводорода с одновременным выделением молекулярного водорода.