ORIGINAL RESEARCH ARTICLE

Preparation of PbTiO₃-CdS nanocomposite material and its microstructure and photocatalytic properties

Mingdong Xu, Wenqiang Li, Shun Liu, Tao Zhang, Sen Lai, Simin Yin*

Faculty of Mechanical Engineering & Automation, Zhejiang Sci-Tech University, Hangzhou 310018, Zhejiang, China. E-mail: yinsm@zstu.edu.cn

ABSTRACT

In order to explore the influence of the ferroelectric surface on the structure and properties of semiconductor oxides, the growth of CdS nanocrystals was regulated and controlled by taking single-crystal perovskite PbTiO₃ nanosheets as the substrate through a simple hydrothermal method. Through composition design, a series of PbTiO₃-CdS nanocomposite materials with different loading concentrations were prepared, and their microstructure and photocatalytic properties were systematically analyzed. Studies show that in the prepared product, CdS nanoparticles selectively grow on the surfaces of PbTiO₃ nanosheets, and their morphology is affected by the exposed surfaces of PbTiO₃ nanosheets. There is a clear interface between the PbTiO₃ substrate and CdS nanoparticles. The concentration of the initial reactant and the time of hydrothermal reaction also significantly affect the crystal morphology of CdS. Photocatalysis studies have shown that the prepared PbTiO₃-CdS nanocomposite material has a significant degradation effect on 10 mg/L of Rhodamine B aqueous solution. The degradation efficiency rises with the increase of CdS loading concentration. When degrading 10 mg/L of Rhodamine B aqueous solution, the PbTiO₃-CdS sample with a mass fraction of 3% can reach a degradation rate of 72% within 120 min.

Keywords: CdS; Single-Crystal Perovskite; PbTiO₃; Nanocomposite Material; Photocatalysis

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1. Introduction

Nano materials have many characteristics, such as small size effect, surface effect, volume effect and unique photoelectric effect, which make them have broad application prospects in energy environment, electronic devices, biomedicine and so on^[1-3]. Perovskite phase PbTiO₃ is a typical ferroelectric oxide material. It was reported by Shirane et al.^[4] in 1950 that the PbTiO₃ is with a Curie temperature of about 480 °C, and with high spontaneous polarization intensity, large dielectric constant and small dielectric loss. It is widely used in sensing, storage, infrared imaging and nano generator devices^[5,6]. With the in-depth study of perovskite materials, researchers have made breakthroughs in the surface research of nanostructures of perovskite materials, and the research of the special surface ferroelectric polarization characteristics has gradually become an important direction^[7]. Using the composite-preparation technology and the ferroelectric polarization characteristics on the surface of perovskite nanostructures can regulate the crystal growth of semiconductor oxides, so as to prepare new nanocomposite materials^[8]. Previous studies have shown that nanocomposite materials have unique electron transport characteristics due to their special energy band structure and surface effects, so they have become a research hotspot in the fields of solar cells, gas-phase catalysis, photocatalysis etc.^[9]

In 1972, Fujishima et al.[10] reported the research on the photolysis of water. In this research, TiO₂ was used as a photoelectric electrode to decompose water under visible light to prepare hydrogen. Semiconductor^[11] was used as an important research object of photocatalytic materials. Researchers have systematically studied other semiconductor oxide systems, such as $g-C_3N_4^{[12,13]}$, ZnO^[14], MoS₂^[15], and Fe₂O₃^[16]. Among many semiconductors, CdS^[17] is a promising photocatalytic material because of its small band gap ($E_g \approx 2.4 \text{ eV}$) and good optical response in the visible band. However, due to its low electron-hole separation efficiency and electron mobility, CdS crystal materials prepared by traditional methods are easy to agglomerate and form large particles, resulting in a significant reduction in their specific surface area, which seriously affects the photocatalytic efficiency of CdS. In view of the above problems, existing studies mainly promote the photocatalytic efficiency of CdS by loading precious metals, such as Pt^[18], Ag^[19], and Au^[20], etc. However, due to the rarity of precious metals and the easy oxidation of CdS, the preparation cost of photocatalytic materials increases.

In this paper, PbTiO₃-CdS nanocomposite materials were successfully synthesized by a secondary hydrothermal method with single-crystal perovskite phase PbTiO₃ nanosheets as substrates and CdCl₂· $5/2H_2O$ and CH₄N₂S as main reactants. Their microstructure and crystal growth were systematically studied. The photocatalytic properties of the prepared PbTiO₃-CdS nanocomposite materials were evaluated by the degradation efficiency of Rhodamine B aqueous solution under ultraviolet light and UV-Vis diffuse reflectance spectrum, which provided experimental material and theoretical basis for the subsequent exploration of the synthesis and properties of more nanocomposite materials.

2. Experiment

2.1 Experimental reagents

Potassium hydroxide (KOH, analytical pure, Hangzhou Gaojing Chemical Co., Ltd.); Titanium dioxide (P25 TiO₂, analytical pure, Shanghai Aladdin Biochemical Technology Co., Ltd.); Lead nitrate (Pb(NO₃)₂, analytical pure, Shanghai Zhanyun Chemical Co., Ltd.); Cadmium chloride hemihydrate (CdCl₂·5/2H₂O, analytical purity); Thiourea (CH₄N₂S, analytical purity) and Rhodamine B (C₂₈H₃₁CIN₂O₃, analytical purity) were provided by McLean Biochemical Technology Co., Ltd.; Absolute ethanol (C₂H₆O, analytical purity, Anhui Ante Food Co., Ltd.); Deionized water (H₂O, self-made in the laboratory).

2.2 Synthesis of materials

2.2.1 Preparation of single-crystal perovskite phase PbTiO₃ nanosheets

Single-crystal perovskite phase PbTiO₃ nanosheets were prepared by the method in the literature^[21]. 0.4 g of TiO₂ powder was added to 6 mol/L of KOH aqueous solution to obtain a solution containing Ti^{4+} with the concentration of 0.1 mol/L. In the stirred state, 0.2 mol/L of $Pb(NO_3)_2$ aqueous solution was added to the Ti⁴⁺-contained solution to adjust the ratio of lead-titanium to 2:0 and stirred for 2 h. The obtained hydroxide solution of titanium and lead was transferred to the inner tank of a 50 mL stainless steel reactor, sealed, and then hydrothermal reaction was carried out at 200 °C for 12 h. After the sample was cooled to room temperature, the sample was centrifuged and cleaned with deionized water and absolute ethanol respectively until the washing solution was neutral. Finally, the washed sample was dried at 60 °C for 24 h to obtain the PbTiO₃ nanosheet sample to be tested.

2.2.2 Preparation of the PbTiO₃-CdS nanocomposite materials

0.2 g of prepared PbTiO₃ nanosheets were dispersed in 30 mL of deionized water to obtain a white suspension. CdCl₂· $5/2H_2O$ and CH₄N₂S were added to the above white suspension at a molar ratio of 1:1 to obtain a mixed solution, which was continuously stirred at room temperature for 30 min. Transfer the above mixed solution to the inner tank of a 50 mL stainless steel reactor, seal it, and then conduct hydrothermal reaction at 160 °C for 12 h. After cooled to room temperature, the sample was centrifuged and cleaned with deionized water and absolute ethanol respectively until the washing solution was neutral. Finally, the washed sample was dried at 60 °C for 24 h to obtain the target product sample to be tested.

2.2.3 Preparation of CdS crystals (contrast samples)

Using the above synthesis method, CdS crystals grown separately were prepared without adding PbTiO₃ nanosheets. The specific synthesis process is as follows: add CdCl₂·5/2H₂O and CH₄N₂S with a molar ratio of 1:1 in 30 mL of deionized water, and obtain the precursor solution by magnetic stirring. After continuous stirring at room temperature for 30 min, transfer the above solution to the inner tank of a 50 mL stainless steel reactor for sealing, and conduct hydrothermal reaction at 160 °C for 12 h. After the reaction, cool the sample to room temperature, centrifuge and clean it with deionized water and absolute ethanol respectively until the eluent was neutral. Finally, the washed sample was dried at 60 °C for 24 h to obtain the contrast product sample to be tested.

2.3 Testing and characterization

2.3.1 Microscopic characterization of materials

The surface morphology of the material was characterized by a Zeiss ULTRA-55 scanning electron microscope (SEM), and the acceleration voltage was 5 kV.

The powder sample was added to the resin with curing agents, stirred and cured by heating. A Leica EM TRIM2 paraffin slicer was used to slice the resin embedded samples. A JEOL-200CX transmission electron microscope (TEM) was used to characterize the fine structure of the materials, and a high resolution transmission electron microscope (HRTEM) was used for imaging analysis, the acceleration voltage of was greater than or equal to 200 kV, the lattice resolution was less than 0.15 nm and the point resolution was 0.21 nm.

2.3.2 Phase analysis of materials

A Shimadzu XRD-6000 X-ray diffractor was

used to carry out phase analysis of materials, with Cu K_a ($\lambda = 0.15406$ nm) as the radioactive source, with a scanning speed of 3 (°)/min and scanning range of 10.0° to 80.0°.

2.3.3 Test of photocatalytic performance

0.2 g of prepared powder sample was dispersed in 50 mL of Rhodamine B aqueous solution (10 mg/L) and dark treated for 30 min to reach adsorption equilibrium. Then, place it in a photochemical reactor (YZ-GHX-A, Shanghai Yanzheng Experimental Instrument Co., Ltd.) and degrade Rhodamine B aqueous solution under UV irradiation of 500 W mercury lamp. Under continuous stirring and illumination, take out 3 mL mixed solution every 20 min, and centrifuge at the speed of 4000 r/min for 5 min to remove the sediment. Measure the absorption peak of the separated supernatant at the wavelength of 554 nm under a UV-Vis gradiometer (UV-1800, Shanghai Mapda Instrument Co., Ltd.). In order to intuitively express the degradation efficiency of Rhodamine B aqueous solution, data processing is carried out through formula (1):

Degradation rate $/\% = C/C_0 \times 100$ (1)

In the formula, C_0 is the initial mass concentration of Rhodamine B aqueous solution, mg/L; C is the mass concentration of the upper clear liquid at an interval, mg/L.

2.3.4 Analysis of UV-Vis diffuse reflectance absorption spectrum

A Shimadzu UV-3600 UV-VIS-NIR spectrophotometer was used for UV-Vis diffuse reflection absorption spectrum analysis. The band gap E_g of *Ahv-hv* image fitted by equation (2):

$$ahv = c(hv - Eg)^2 \tag{2}$$

In the formula, α is the absorbance coefficient, which is directly proportional to absorbance A, and it does not affect the fitting result of the band gap width E_g , so in the final image, α is replaced by A, c is a constant. hv is obtained from equation (3):

 $hv = (h \times c)/\lambda \tag{3}$

In the formula: *h* is Planck constant, $h = 6.63 \times 10^{-34}$ J; *c* is the speed of light, taken as 3×10^8 m/s; the unit of *hv* is J. According to $1 \text{ eV} = 1.6 \times 10^{-19}$ J, the obtained *hv* was converted to eV.

3. Results and analysis

3.1 Analysis of the morphology of the PbTiO₃-CdS nanocomposite materials

Figure 1(a) is the SEM diagram of the perovskite phase PbTiO₃ sample prepared by the hydrothermal method. As can be seen from Figure 1(a), the PbTiO₃ sample is a sheet square with a side length of about 600 nm and a thickness of about 100 nm, with smooth surface, regular morphology and good dispersion. Its large-area exposed surface is (001) crystal surface^[21]. Figure 1(b) shows the contrast sample, that is the morphology of CdS particles grown alone without PbTiO₃ nanosheet substrate. According to Figure 1(b), the CdS particles grown alone were of micron structure with leaf and branch morphology with size of $2-3 \mu m$. The surface is smooth and flat, with clear particle contour and aggregation tendency. Figures 1(c)-(e) are SEM images of samples with different CdS loading concentrations. As can be seen from Figure 1(c), most of the PbTiO₃ nanosheets in the sample with PbTiO₃-CdS mass fraction of 1% have smooth surface and no obvious adhesion of CdS nanoparticles. It can be seen from Figure 1(d) that more CdS nanoparticles with a size of about 10 nm are grown on the (001) crystal surface of PbTiO₃ nanosheets in the sample with PbTiO₃-CdS mass fraction of 2%. Figure 1(e) shows that in the sample with PbTiO₃-CdS mass fraction of 3%, CdS is not only dispersed on the (001) crystal plane of PbTiO₃, but a small amount of CdS is selectively dispersed on the side of $PbTiO_3$ nanosheets, that is, the (100) or (010) crystal plane. With the increase of CdS loading concentration, the amount of particles grown on PbTiO₃ nanosheets gradually increases, and its growth crystal surface has obvious selectivity, which may be related to the polarity of the exposed crystal surface of PbTiO₃ nanosheets. Under different initial reactant additions, it can also be observed that there is crystal plane selectivity in the growth of CdS nanoparticles on PbTiO₃ nanosheets, that is, it is preferred to nucleate and grow on the strongly polarized plane, and then composite on other crystal planes.





(c) PbTiO₃-CdS with a mass fraction of 1% (d) PbTiO₃-CdS with a mass fraction of 2% (e) PbTiO₃-CdS with a mass fraction of 3%

Figure 1. SEM images of single-crystal perovskite phase PbTiO₃ nanosheets synthesized by the hydrothermal method, CdS particles and PbTiO₃-CdS nanocomposite materials with different CdS loading concentrations.

3.2 Phase analysis of PbTiO₃-CdS nanocomposite materials

Figure 2(a) shows the XRD pattern of perovskite phase PbTiO₃ nanosheets prepared by the hydrothermal method, with all corresponding diffraction peaks (JCPDS: 06-0452), sharp diffraction peaks, good crystallinity and no impurity diffraction peaks. The diffraction peak intensity of its (001) crystal plane is almost equal to the highest peak (101) of the standard diffraction pattern, indicating that the prepared PbTiO₃ sample has a large number of (001) exposed surface, which is consistent with the observation results reported in the literature^[21] and **Figure 1(a)**. **Figure 2(b)** shows the XRD patterns of CdS particles grown alone and samples with different CdS loading concentrations. It can be seen from the figure that the diffraction peaks of CdS particle samples synthesized separately all correspond to the standard PDF card (JCPDS: 41-1049), with sharp diffraction peaks and no impurity diffraction peaks. The diffraction peaks of PbTiO₃-CdS with a mass fraction of 1%, 2% and 3% are obvious, and the diffraction of PbTiO₃ is more intense, and the diffraction peak of CdS is relatively weak. With the increase of CdS loading concentration, the diffraction peak is gradually obvious. It is consistent with the SEM results in **Figures 1(c)** to (e). And in the XRD diffraction pattern of PbTiO₃-CdS nanocomposite materials, the diffraction peak intensity corresponding to the (001) crystal plane in PbTiO₃ (JCPDS: 06-0452) decreases, which may be related to the selective growth of CdS on the (001) crystal plane of PbTiO₃ nanosheets.



Figure 2. XRD patterns of single-crystal perovskite phase PbTiO₃ nanosheets synthesized by the hydrothermal method, single-synthesized CdS particles and PbTiO₃ CdS nanocomposite materials with different CdS loading concentrations.

3.3 Microstructure analysis of PbTiO₃-CdS nanocomposite materials

In order to further explore the growth and morphology of CdS nanoparticles on the surface of perovskite PbTiO₃ nanosheets, the resin embedded sections of PbTiO₃-CdS nanocomposite materials were characterized and analyzed by TEM and HRTEM. As shown in **Figure 3(a)**, CdS nanoparticles are dispersed on the upper and lower crystal planes of PbTiO₃ nanosheets, and an obvious lattice interface is formed, as shown in **Figure 3(b)**. The nanoparticle has complete outline and clear lattice, and its crystal plane spacing is 0.316 nm and 0.245 nm respectively, which corresponds to the (101) and (102) crystal planes of CdS (JCPDS: 41-1049). The crystal plane spacing of PbTiO₃ nanosheets is 0.415 nm and 0.390 nm respectively, which corresponds to the (001) and (100) crystal planes of PbTiO₃ (JCPDS: 06-0452). Therefore, it is further proved that the prepared samples are PbTiO₃-CdS nanocomposite materials, and CdS nanoparticles grow selectively on the surface of PbTiO₃ nanosheets.



(a) TEM

(b) HRTEM

Figure 3. TEM and HRTEM diagrams of PbTiO₃-CdS nanocomposite materials synthesized by the hydrothermal method.

3.4 Growth process of PbTiO₃-CdS nanocomposite materials

In order to further explore the growth process PbTiO₃-CdS nanocomposites, PbTiO₃-CdS of nanocomposite materials grown under different hydrothermal reaction time were analyzed. Figure 4 and Figure 5 show the SEM and XRD results of the prepared samples respectively. As can be seen from Figure 4: (a) no obvious CdS nanoparticles were observed on the surface of PbTiO₃ nanosheets in the 0.5-h sample, and there was no corresponding diffraction peak in XRD. At this time, CdS had not crystallized because the hydrothermal time was too short. (b) Fine CdS nanoparticles with a size of about 10 nm have appeared on the surface of PbTiO₃ nanosheets in the 3-h sample. Comparing the XRD pattern of this sample with that of the 0.5-h sample, it can be found that the diffraction peak corresponding to CdS gradually strengthen. (c) The CdS nanoparticles grown on the surface of PbTiO₃ nanosheets in the 5-h sample gradually increased, and grew on each surface. Compared with the XRD pattern of 3-h sample in (b), the diffraction peak corresponding to CdS in this sample pattern was enhanced again. With the extension of the reaction time to 7 h in (d), the size and quantity of CdS nanoparticles on the surface of PbTiO₃ nanosheets increase. At this time, CdS nanoparticles can be obviously observed on some surfaces of PbTiO₃ nanosheets, with smooth surfaces and no

particle adhesion. Therefore, it can be seen that the growth of CdS on the surface of PbTiO₃ nanosheets appears selectivity under the growth conditions. With the further extension of the reaction time (Figures 4(e)-(f)), the size of CdS nanoparticles on the surface of PbTiO₃ nanosheets remains stable, and its growth selectivity is more obvious. Statistically, a large number of blank exposed crystal surfaces can be observed, while a large number of CdS particles grow on other crystal surfaces. Figure 5 shows the XRD diffraction results of the prepared samples. It can be seen from the figure that the reaction products have sharp diffraction peaks, which can correspond to two substances of perovskite phase PbTiO₃ (JCPDS: 06-0452) and CdS (JCPDS: 41-1049) on the standard PDF card. The diffraction results show that both PbTiO₃ substrate and CdS grown on its surface have good crystallinity. According to the above analysis results, with the extension of the reaction time, the growth of CdS nanoparticles on PbTiO₃ nanosheets was significantly regulated. CdS nanoparticles selectively grow on a crystal surface of PbTiO₃ nanosheets. With the increase of the hydrothermal time, according to the crystallization thermodynamics, the size of CdS nanoparticles gradually increases and stabilizes. The growth of CdS on the surface of PbTiO₃ nanosheets is a process regulated by thermodynamics and exposed crystal surface of PbTiO₃.



(a) 0.5 h

(b) 3 h

(c) 5 h



(d) 7 h (e) 9 h (f) 12 h Figure 4. SEM of PbTiO₃-CDs nanocomposite materials synthesized under different hydrothermal reaction time.



Figure 5. XRD patterns of PbTiO₃-CdS nanocomposite materials synthesized under different hydrothermal reaction time.

3.5 Study on the photocatalytic properties of PbTiO₃-CdS nanocomposite materials

Based on the above research, the photocatalytdegradation properties of the prepared ic PbTiO₃-CdS nanocomposite materials were studied. Figure 6 shows the efficiency curves of self-degradation of Rhodamine B solution and degradation Rhodamine B solution by different samples under UV irradiation. As shown in Figure 6, Rhodamine B solution has slight self-degradation within 120 min, and the degradation rate is 10%, which is caused by the slight increase of the temperature of the stirred solution under UV irradiation in the experiment. PbTiO₃ nanosheets and PbTiO₃ nanosheets + CdS particles have almost the same degradation ability to Rhodamine B solution, and the degradation efficiency is low, which is 40%. It shows that the photocatalytic activity of PbTiO₃ nanoparticles and CdS particles is not high. The Rhodamine B solution was significantly degraded by PbTiO₃-CdS with a mass fraction of 1%, 2% and 3% under UV light, and the degradation rates were 60%, 64% and 72% respectively. The above results show that with the increase of CdS loading, the catalytic degradation efficiency of PbTiO₃-CdS nanocomposite materials on Rhodamine B aqueous solution increases significantly. The degradation efficiency of PbTiO₃-CdS with a mass fraction of 3% is 1.8 times higher than that of PbTiO₃ nanosheets and PbTiO₃ nanosheets + CdS particles.



Figure 6. Efficiency curves of self-degradation of Rhodamine B solution and degradation by different samples.







Figure 8. Ahv-hv fitting image.

Figure 7 shows the UV-Vis diffuse reflectance spectra of different samples. It can be seen from the figure that the prepared PbTiO₃-CdS nanocomposite materials have obvious absorption peaks between 425 nm and 520 nm, indicating that PbTiO₃-CdS nanocomposite materials have good response to visible light. With the increase of CdS loading concentration, the response of PbTiO₃-CdS nanocomposite materials to visible light increased slightly. The *Ahv-hv* fitting image of each sample is fitted

according to formula (2), as shown in Figure 8, and the band gap width of each sample is shown in Table 1. The two band gap widths of PbTiO₃-CdS nanocomposite materials not only improve the band gap width of PbTiO₃ nanosheets, but also improve the band gap width of CdS particles ($E_g \approx 2.4 \text{ eV}$), so as to reduce the energy required for electronic transition and improve the photocatalytic performance. With the increase of CdS loading concentration to 3%, the light absorption of PbTiO3-CdS composite samples with a mass fraction of 3% in 425-520 nm band increases significantly. The increase of light absorption is conducive to the formation of electrons and holes in the composites. The absorption edge of PbTiO₃-CdS composite samples with a mass fraction of 3% also has a small amount of red shift, indicating that the band gap of the sample is also reduced accordingly, which is conducive to the absorption of small energy photons, promoting the generation and separation of electrons and holes, and further improving the photocatalytic performance of the sample. This is also consistent with the fact that PbTiO₃-CdS samples with a mass fraction of 3% show the highest photocatalytic activity on Rhodamine B aqueous solution under light. Based on the above analysis, a possible photocatalytic mechanism is proposed: perovskite PbTiO₃ nanosheets and CdS are used as light absorption centers to absorb photon energy and produce photogenerated carriers respectively. The CdS surface is the photocatalytic reaction center. The built-in electric field formed by the spontaneous polarization of PbTiO₃ nanosheets promotes the separation of electrons and holes, and the electrons are further transferred to the CdS surface for photocatalytic reaction, so as to improve the photocatalytic effect of PbTiO₃-CdS.

Samples	Band gap width 1/eV	Band gap width 2/eV
PbTiO ₃ nanosheets		2.76
PbTiO ₃ -CdS with a mass fraction of 1%	2.24	2.67
PbTiO ₃ -CdS with a mass fraction of 2%	2.21	2.52
PbTiO ₃ -CdS with a mass fraction of 3%	2.21	2.46

Table 1. Band gap width of PbTiO₃ nanosheets and PbTiO₃-CdS with different loading concentrations

4. Conclusion

In this paper, PbTiO₃-CdS nanocomposite materials were successfully prepared by the hydrothermal method, and their microstructure and crystal growth process were systematically characterized and analyzed. On this basis, the photocatalytic properties of the prepared PbTiO₃-CdS nanocomposite materials were evaluated. The main conclusions are as follows:

a) The prepared PbTiO₃-CdS nanocomposite materials have regular morphology and good dispersion; CdS nanoparticles (about 10 nm in size) were selectively grown on the surface of perovskite PbTiO₃ nanosheets with good crystallinity. Compared with CdS particles with independent nucleation and crystallization, its size decreases sharply, and an obvious interface is formed with PbTiO₃ nanosheets. The crystal growth of PbTiO₃-CdS nanocomposite materials is a process regulated by the exposed crystal surface of PbTiO₃.

b) Hydrothermal reaction time is an important thermodynamic condition that strongly affects the crystal growth process of PbTiO₃-CdS nanocomposite materials. With the increase of time, the more sufficient the nucleation and crystal growth of CdS nanoparticles on the surface of PbTiO₃ nanosheets, the larger the CdS grain size.

c) The prepared PbTiO₃-CdS nanocomposite materials can degrade 10 mg/L Rhodamine B solution. With the increase of the mass fraction of PbTiO₃-CdS, the photocatalytic degradation efficiency of PbTiO₃-CdS nanocomposite materials increases. The degradation rate of PbTiO₃-CdS nanocomposite materials with a mass fraction of 3% was the highest at 120 min, reaching 72%.

Conflict of interest

The authors declare that they have no conflict of interest.

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