

Research Article

# Enhanced photocatalytic activity of silver vanadate nanobelts in concentrated sunlight delivered through optical fiber bundle coupled with solar concentrator



Joy Sankar Roy<sup>1</sup> · Gabriel Dugas<sup>2</sup> · Steeve Morency<sup>2</sup> · Sidney J. L. Ribeiro<sup>1</sup> · Younès Messaddeq<sup>1,2</sup>

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### **Abstract**

To date, sunlight driven photocatalytic process for wastewater treatment is a great challenge. Herein, the photocatalytic methylene blue (MB) dye degradation activity of silver vanadate nanobelts has been enhanced using concentrated sunlight irradiation. The MB dye degradation factor in normal and concentrated sunlight irradiation is 0.57 and 0.25 respectively after 120 min of light exposure. Therefore, degradation of MB dye in concentrated sunlight occurs more than two times faster than normal sunlight. The silver vanadate nanobelts have been prepared by simple hydrothermal method. The prepared nanobelts are very thin having length ranging between 5 and 10 µm and width is in the 100–300 nm range. The optical band gap of the synthesized silver vanadate nanobelts is 1.96 eV (632.5 nm), which indicates strong absorption of visible light. This work will open new technological aspects for cost-effective sustainable wastewater treatment using solar energy.

 $\textbf{Keywords} \ \ \textbf{Silver vanadate} \cdot \textbf{Sunlight driven photocatalysis} \cdot \textbf{Concentrated sunlight} \cdot \textbf{Solar concentrator} \cdot \textbf{Optical fiber} \cdot \textbf{Water purification}$ 

# 1 Introduction

Water is an essential constituent for the survival of human beings, although millions of people worldwide are suffering from lack of fresh and clean drinking water. The ground water and surface water have been polluted by rapid speed of industrialization, expansion of population, unplanned urbanization and environmental pollution [1–4]. The discharge of untreated sanitary and toxic industrial wastes, dumping of industrial effluent, and run off from agricultural fields are the main sources of water pollution [5, 6]. The agriculture and pharmaceutical effluents release pesticides and other chemicals that are responsible for some chronic diseases [5–8]. Various industries such as textiles, dyeing and printing discharge large amounts

of synthetic organic dyes as effluents. According to the World Health Organization's (WHO) 2017 report, around 844 millions of people worldwide have no scope to utilize fresh and clean water. In developing countries, almost 70% of all the illness are related to water contamination [9, 10]. Therefore, it is the greatest challenges in twenty-first century for the researchers to develop an eco-friendly, cost-effective and fast processing technology to remove the pollutants from wastewater in order to protect the environment and human beings. To address this serious issue, semiconductor photocatalysis has been believed as a low cost, sustainable and environmentally friendly approach by making use of solar energy [11].

The semiconductor photocatalysts have been studied extensively for purification of wastewater. Among the

<sup>☑</sup> Joy Sankar Roy, jsr.phys@gmail.com; ☑ Younès Messaddeq, younes.messaddeq@copl.ulaval.ca | ¹Institute of Chemistry, Sao Paulo State University (UNESP), Araraquara, SP 14800-060, Brazil. ²Center for Optics, Photonics and Lasers (COPL), Laval University, Quebec, QC G1V 0A6, Canada.



semocinductor photocatalysts, titanium dioxide (TiO<sub>2</sub>) is widely used owing its outstanding photocatalytic activity, non-toxicity and high photostability [12-14]. But, TiO<sub>2</sub> is only sensitive under UV-light since it has large bandgap energy of 3.2 eV and utilizes only 4% of available solar energy, which highly restricts its potential application as photocatalyst [14-17]. Therefore, numerous efforts have been made to enhance photocatalytic activity of TiO<sub>2</sub> in visiblelight, including anion doping, metal doping, and oxygen deficiency creation [18–20]. Although the light absorption of doped TiO<sub>2</sub> was extended to visible light region, these processes often suffered from thermal instability or carrierrecombination [21, 22]. These methods are not appropriate to develop highly efficient visible-light active photocatalysts. Hence, researchers have devoted much attention to fabricate visible-light driven photocatalysts to enhance the use of solar energy in the process of wastewater purification.

Recently, researchers have developed band structure control technique to fabricate non-TiO<sub>2</sub>-based visible-light active photocatalysts, such as BiVO<sub>4</sub> [11], AgNbO<sub>3</sub> [23], Bi<sub>2</sub>WO<sub>6</sub> [24] Ag<sub>3</sub>VO<sub>4</sub> [25], AgVO<sub>3</sub> [25], CoFe<sub>2</sub>O<sub>4</sub> [26], Zr<sub>2</sub>Ni<sub>2</sub>Cu<sub>7</sub> [27]. Among thses photocatalysts, silver vanadate has drawn immense interest due to their excellent antibacterial, electrochemical and photophysical properties along with photocatalytic activity [25, 28, 29]. The researchers are still trying to enhance photocatalytic activity of silver vanadate nanomaterials using various techniques [30–33]. In this work, we report the synthesis of silver vanadate nanobelts by a simple hydrothermal method without using any template or surfactant and their photocatalytic activity in the degradation of Methylene Blue under normal and concentrated sunlight irradiation. We have used a solar concentrator coupled with optical fiber bundle to deliver concentrated sunlight to the photocatalytic reactor. Most of the researchers use Xenon arc lamp or fluorescent lamp or LED lamp or solar simulator as visible light source to study photocatalytic activity [23-25, 34, 35]. Recently, some of them studied photocatalysis process in direct sunlight irradiation [26, 27, 36]. This is the first time in literature we are using a novel technology to use concentrated sunlight as visible light source to study photocatalytic activity. The utilization of solar concentrator coupled optical fiber bundle as the concentrated sunlight source provides a promising direction for future applications of low cost and fastest sustainable wastewater purification process.

# 2 Experimental

### 2.1 Preparation of photocatalysts

Silver vanadate nanobelts were prepared using simple hydrothermal method. In a typical procedure, 2 mmol of  $\rm NH_4VO_3$  and 2 mmol of AgNO $_3$  was dissolved in 40 mL distilled water seperately and then AgNO $_3$  aqueous solution was mixed dropwise into  $\rm NH_4VO_3$  aqueous solution under magnetic stirring. The resulting suspension was transferred into a 100 mL Teflon-lined stainless steel autoclave and kept at 150 °C for 4 h and then cooled to room temperature. The resulting sample was collected and washed with distilled water and ethanol several times and dried overnight at 70 °C.

### 2.2 Sample characterization

The morphology of the prepared sample was investigated using field emission scanning electron microscope (JEOL, JSM 7500F) and transmission electron microscope (JEOL, JEM 1230). Structural properties were studied using Selected Area Electron Diffraction (SAED) and X-ray diffraction (XRD). The XRD pattern of the powder silver vanadate nanobelts was studied using Bruker D8 Advance X-ray diffractometer. Raman spectroscopy was recorded using Horiba Jobin-Yvon (HR 800) Micro-Raman under 632.8 nm laser irradiation. The Raman spectrum was recorded in the 100–1200 cm<sup>-1</sup> wavenumber range. The optical absorption characteristic of the sample was investigated using the Varian Cary 5000 UV-Vis-NIR spectrophotometer. Optical power of normal and concentrated sunlight was measured using Gentec-eo UP55N-300F-H12-D0 power meter. An agilent 1200 Liquid Chromatography Mass Spectrometer (LCMS) was used to identify the intermediates of MB dye during degradation.

### 2.3 Photocatalytic activity evaluation

The photocatalytic activity of silver vanadate nanobelts was investigated by degradation of MB dye under normal and concentrated sunlight irradiation. The concentrated sunlight has been delivered to photocatalytic reactor through optical fiber bundle coupled with solar concentrator. The used solar concentrator is shown in Fig. 1. It consists main four parts: optical reflector, optical fiber bundle, solar tracker and mechanical support. The optical reflector system consists of two mirrors: primary parabolic mirror and secondary flat mirror. Primary mirror captures sunlight and projects concentrated sunlight on secondary mirror. Secondary mirror projects concentrated sunlight on the top of the optical fiber bundle. The optical fiber are made with pure silica to tolerate high temperature at the focal point of the concentrated sunlight and also to pass UV light. The solar tracker is fixed on the arm of the optical system so that it can track the position of the sun and moves according to the movement of the sun. Photocatalytic reactor consists of two small fans to circulate fresh air, a magnetic stirrer to stir photocatalysts suspended MB dye

Fig. 1 Parabolic mirror based solar concentrator coupled with optical fiber bundle



solution, and an optical fiber bundle covered with pure silicate glass to deliver concentrated sunlight from solar concentrator. The photocatalytic reactor is shown by magnification in Fig. 1.

For photocatalytic degradation of MB dye, 20 mg of silver vanadate was dispersed in 50 ml of 20 ppm MB dye solution under ultrasonication for 2 min, and was kept in the dark place for 2 h to achieve adsorption–desorption equilibrium. At regular intervals of time, aliquots of the suspension were collected and analyzed in UV–Vis–NIR spectrophotometer (Varian Cary 5000).

## 3 Results and discussion

### 3.1 Morphology and structure

The morphology of the silver vanadate photocatalysts was observed in FESEM and is ahown in Fig. 2 with different magnification. Figure 2 reveals the high yield and homogeneity of nanobelts structure. The SEM images confirm that no other structure except nanobelts are formed. It can be seen in Fig. 2, that length of the nanobelts ranging between 5 and 10  $\mu m$  and width is in the 100–300 nm range, and the thickness is in order of few nm. The size of

the nanobelts can be better understood from TEM images as shown in Fig. 3. The TEM image of a nanobelt is shown in Fig. 3a and 3b. The length and width of the shown nanobelt is 7  $\mu$ m and 152 nm respectively.

The SAED was studied to conform the crystalline nature of the silver vanadate nanobelts. The SAED pattern is shown in Fig. 3c. The diffused concentric circles and bright spots on SAED image signifies well polycrystalline nature of the silver vanadate nanobelts. Figure 4 displays the XRD pattern of the prepared silver vanadate nanobelts. All the diffraction peaks of the as-synthesized sample conform to pure  $\beta$ -AgVO<sub>3</sub> phase (space group: C12m1, 56861-ICSD) [37]. The (612) peak is the strongest one, indicationg possible preferential orientation of  $\beta$ -AgVO<sub>3</sub> nanobelts. The XRD pattern confirms the non-exsistence of  $\alpha$ -AgVO<sub>3</sub> phase or silver in the prepared sample.

Figure 5 shows the Raman spectrum of the prepared silver vanadate nanobelts. The Raman band at 956 cm<sup>-1</sup> is attributed to symmetric stretching of VO<sub>4</sub> units and the strong band at 886 cm<sup>-1</sup> originates from either bridging V–O–Ag or O–V–O vibrations [37, 38]. The bridging Ag–O–Ag bond is reflected by 808 cm<sup>-1</sup> Raman band. The Raman bands at 732 cm<sup>-1</sup> and 519 cm<sup>-1</sup> are associated with asymmetric and symmetric stretches of V–O–V bond, respectively. The Raman bands at 336 cm<sup>-1</sup> and 386 cm<sup>-1</sup>

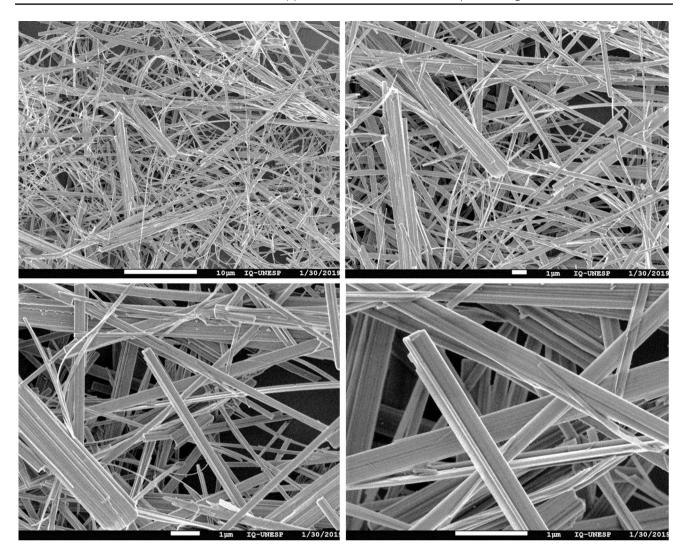


Fig. 2 FESEM images of the  $\beta\text{-AgVO}_3$  nanobelts with different magnification

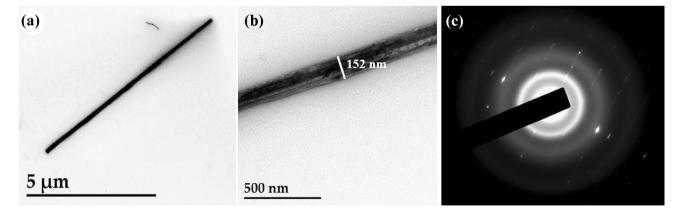


Fig. 3 a, b TEM images and c corresponding SAED pattern of the  $\beta$ -AgVO<sub>3</sub> nanobelts

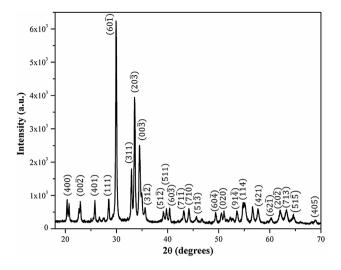


Fig. 4 XRD pattern of the as-prepared silver vanadate nanobelts

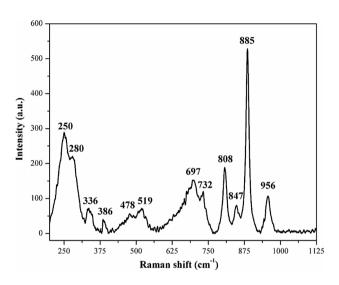
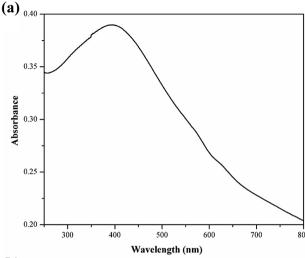
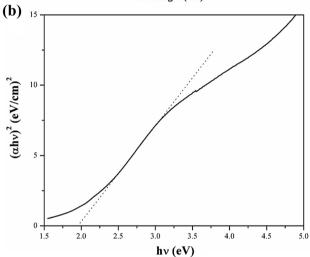


Fig. 5 Raman spectrum of the as-prepared silver vanadate nanobelts

correspond to the asymmetric and symmetric deformation modes of the  $VO_4^{3-}$  tetrahedron, respectively [37]. The symmetric and asymmetric bending modes of the  $VO_4$  units occur at 280 cm<sup>-1</sup> and 250 cm<sup>-1</sup>, respectively [38].





**Fig. 6 a** UV–visible absorption spectrum and **b** Tauc plot of the asprepared silver vanadate

The Raman spectrum reflects the pure  $\beta$ -AgVO<sub>3</sub> phase of the prepared sample [37, 38].

# 3.2 Optical properties

The UV-visible absorption spectrum of the prepared silver vanadate nanobelts is shown in Fig. 6a. The absorption

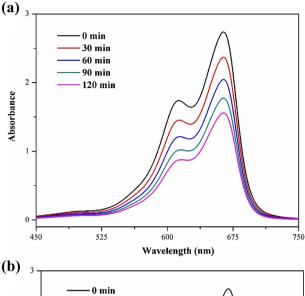
**Table 1** Variation of solar irradiance power with wavelength for normal and concentrated sunlight

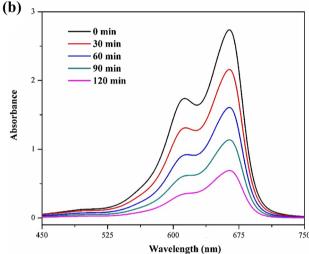
Wavelength (nm)	Irradiance power (mW/cm²)	
	Normal sunlight	Concen- trated sunlight
632	73.9	324.9
532	72.3	318.6
488	71.5	315.1

band shows maximum at 400 nm, but the absorption edge starts from 650 nm. Therefore, the prepared nanobelts are capable to absorp light in wide range of visible region. To better understand the absorption of optical energy, optical band gap has been calculated using Tauc plot [39–42] as shown in Fig. 6b. The estemated optical band gap is 1.96 eV. Therefore, Fig. 6 reveals that the prepared silver vanadate nanobelts are very much sensitive in visible light.

# 3.3 Photocatalytic activity

Photocatalytic activity of silver vanadate nanobelts has been studied under direct or natural sunlight and concentrated sunlight irradiation. The irradiance power of used concentrated sunlight is much higher that the normal sunlight. The irradiance power of normal and concentrated sunlight is summarised in Table 1. Though the optical band gap of the silver vanadate nanobelts is 632 nm (1.96 eV), we have measured solar irradiance power for different wavelength like 488 nm, 532 nm and 632 nm because the nanobelts show broad absorption spectrum as shown in Fig. 6. The irradiance power of concentrated sunlight has been measured at distance 18 cm from the optical fiber bundle where the sample was kept for experiment. Photocatalytic activity of the silver vanadate nanobelts for degradation of MB dye under sunlight is shown in Fig. 7. Figure 7a represents comparision of absorbance spectra of MB dye with duration of normal sunlight exposure and similar activity for concentrated sunlight is shown in Fig. 7b. It can be seen from Fig. 7, that the absorbance peak of MB dye decreases gradually with duration of sunlight exposure and it occurs faster for concentrated sunlight. The photocatalytic activity of the silver vanadate nanobelts can be better understood from Fig. 8. Relative degradation of the MB dye is shown in Fig. 8a and time





**Fig. 7** Comparision of absorption spectra of MB dye after irradiation of **a** normal sunlight and **b** concentrated sunlight

dependent degradation efficiency is shown in Fig. 8b. Relative degradation is expressed by  $C/C_0$ , where C is dye concentration after certain duration of light exposure and  $C_0$  is the initial dye concentration. It is clear from Fig. 8 that the degradation occurs faster for concentrated light. The degradation factor for normal and concentrated sunlight is 0.57 and 0.25 respectively after 120 min of light exposure. Therefore, degradation of MB dye in concentrated sunlight is more than two times faster than normal sunlight. The enhancement in MB dye degradation under concentrated sunlight can be clearly understood from

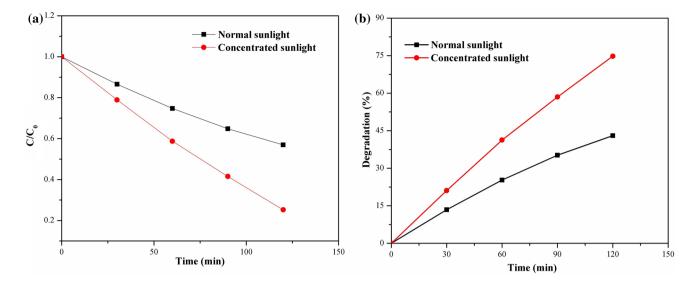


Fig. 8 a Relative degradation and **b** corresponding % degradation of MB dye catalized by as-synthesized silver vanadate nanobelts under normal and concentrated sunlight irradiation

Fig. 8b. Silver vanadate nanobelts are capable to degrade 75% of MB dye just within 120 min of concentrated sunlight exposure, where as only 43% degradation occurs for normal sunlight exposure. Recently, Gonzalez-Zavala et al. reported 37% degradation efficiency for Malachite green dye using photocatalytic activity of silver vanadate thin film and they achieved maximum of 53% degradation efficiency by doping silver with silver vanadate thin film after 180 min exposure of light [34]. They used solar simulator to deliver artificial sunlight for photocatalytic activity. Belver et al. reported around 40% degradation of Rhodamine B dye in 420 min using AgVO<sub>3</sub> nanoparticles [35]. In our work, we got 75% degradation efficiency for MB dye using concentrated sunlight just within 120 min. Therefore, solar concentrator coupled with optical fiber bundle will open up new technological aspect to utilize solar energy for wastewater treatment.

### 3.4 Photocatalysis mechanism

The prepared silver vanadate nanobelts absorb sunlight with energy equal to or greater than the band gap energy (1.96 eV) of the silver vanadate and generate electrons and holes in the valence and conduction band respectively. The generated electrons and holes migrate to the surface of the silver vanadate nanobelts and participate

in oxidation and reduction process to produce hydroxyl and superoxide radicals respectively. The formed hydroxyl and superoxide radicals react with MB dye to degrade it. The MB dye is degraded to harmless or less-harmful products like carbon dioxide ( $CO_2$ ), water ( $H_2O$ ) etc. through some intermediates compounds. These intermediates compunds were identified by LCMS and is shown in Fig. 9. Based on the ratios of mass to charge (m/z) in the mass spectra (Fig. 9), the corresponding compounds and their structural formulas have been deduced and the photocatalytic degradation pathway of MB dye (m/z=284) has been proposed in Fig. 10.

Based on our experimental results and earlier reports in literature on photocatalytic degradation of organic molecules, photocatalytic degradation mechanism for MB dye using  $\beta$ -AgVO<sub>3</sub> photocatalyst is proposed as follows [43, 44]:

$$AgVO_3 + h\nu \rightarrow AgVO_3(e^- + h^+)$$

$$h^+ + H_2O \rightarrow \cdot OH$$

$$h^+ + OH^- \rightarrow \cdot OH$$

$$e^- + O_2 \rightarrow \cdot O_2^-$$

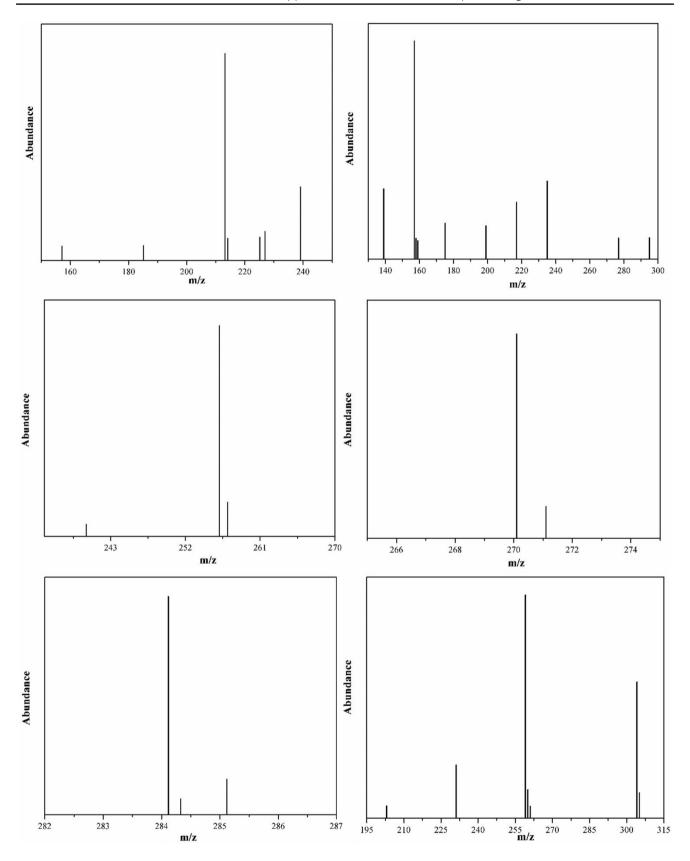


Fig. 9 LC-MS spectra of MB during photocatalytic degradation process

Fig. 10 Possible degradation pathway of MB during photocatalysis

 $MB + \cdot OH \rightarrow intermediates \rightarrow degradation products$ 

 $MB + \cdot O_2^- \rightarrow intermediates \rightarrow degradation products$ 

Photocatalytic activity of silver vanadate nanobelts enhances in presence of concentrated sunlight because concentrated sunlight has more irradiance power and contains more number of photons. These excess photons create more electron–hole pairs and hence more hydroxyl and superoxide radicals are formed to degrade MB dye faster.

### 4 Conclusions

The nanobelts of  $\beta$ -AgVO $_3$  have been prepared by simple hydrothermal method. The prepared thin nanobelts are of well crytalline and uniform having length between 5 and 10  $\mu$ m with width 100–300 nm. These nanobelts have high capacity to absorb visible light and have optical band gap 1.96 eV. The  $\beta$ -AgVO $_3$  nanobelts exhibited high visible-light photocatalytic activity in the degradation of Methylene Blue dye under sunlight irradiation. The dye degradation rate becomes more than two times faster under concentrated sunlight irradiation. The concentrated sunlight has been utilized to degrade MB dye using solar concentrator coupled with optical fiber bundle. This new technology can be implemented to purify industrial wastewater using solar energy in a sustainable and cost-effective way.

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## Compliance with ethical standards

**Conflict of interest** On behalf of all authors, the corresponding author states that there is no conflict of interest.

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