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Green synthesis of Ag nanoparticles based on Moringa Oleifera to increase organic dye D205 absorbance on ZnO nanorod electrode

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ABSTRACT

Green synthesis of AgNPs using Moringa Oleifera extract has many advantages, including low toxicity, relatively low cost, environmentally friendly materials, and the availability of easily obtainable materials. AgNPs also have the advantage of enhancing light absorption in the visible region due to the occurrence of an optical phenomenon known as Localized Surface Plasmon Resonance (LSPR). In this study, AgNPs were composited with the organic dye D205 to examine the effect of AgNPs composites on the increased absorbance of the organic dye D205 on ZnO nanorod electrodes and its influence on the material's band gap. The method used in this research is the green synthesis of AgNPs by reducing Moringa Oleifera extract with the reducer $AgNO_3$, which produces AgNPs powder. Then, in this study, ZnO nanorod electrodes were synthesized using the hydrothermal method. After that, the ZnO nanorod electrode was immersed with the organic dye D205, which was composited with AgNPs. The variation in this study lies in the variation of AgNPs composites at 0 wt% and 10 wt%. Sample characterization was performed using XRD, SEM, and UV-Vis. This study shows that the crystal size of AgNPs is 34 nm, and the maximum diffraction peak is at $2\theta = 32.16^{\circ}$ on the hkl (122) plane. The SEM results show an average diameter of AgNPs of 85 nm, with EDX indicating an atomic weight of Ag compound of 79%. The absorbance values in this study from the variations of AgNPs composites and organic dyes at 0 wt% and 10 wt% were 321 nm and 325 nm, respectively. The Band gap values of the AgNPs and organic dye composite variations at 0 wt% and 10 wt% are 3.69 eV and 2.99 eV, respectively. The research results show that the AgNPs composite with the organic dye D205 successfully increased absorbance and reduced the material's band gap by about 0.7 eV. Thus, the AgNPs composite with the organic dye D205 has the potential to improve efficiency in ZnO nanorod-based DSSC applications.

Keywords: AgNPs, D205, *Green synthesis*, ZnO nanorod

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INTRODUCTION

The development of nanotechnology is currently progressing rapidly, especially in the field of science (Razavi et al., 2021). Thus, various types of materials can be synthesized into nanoparticles sized 1 – 100 nm using various methods (Tesfaye et al., 2023). Metal nanoparticles can be made to non-metric sizes using destructive or constructive processes.

Metals such as aluminium (Al), copper (Cu), gold (Au), iron (Fe), silver (Ag), and zinc (Zn), which are some types of metals commonly used for nanoparticle synthesis (Pattanayak et al., 2021). Silver nanoparticles (AgNPs) in nanotechnology play an important role due to their diverse applications in various fields, including biology, optics, catalysis, and health (C K et al., 2024). AgNPs can be synthesized using the biosynthesis method (Gahlawat & Choudhury, 2019). Synthesis using the biosynthesis method on AgNPs can control the nanoparticle size within AgNPs crystals (Amiri et al., 2018). Biomolecules in the plant extracts used play an important role in the formation of nanoparticle size (Amiri et al., 2017). AgNPs are a type of transition metal suitable for use as doping or composite in a material because they have high electrical conductivity and good light absorption capabilities (Hosseini et al., 2015).

AgNPs are composited with the organic dye D205 because it has advantages that can significantly enhance the light absorption process. Organic dyes are important components that act as photosensitizers to enhance cell performance and strengthen light absorption (Miao et al., 2022). Organic dyes have a relatively low cost and a high excitation coefficient, allowing electrons to pass through the conductive substrate to generate electric current (Kusumawati et al., 2021). Organic dyes such as D205 based on Diphenylethenyl can achieve an efficiency of around 1.62% due to their high charge transfer capability and absorption intensity in the visible light region, specifically at wavelengths of 400 nm-700 nm (De & Niyas, 2023) as components in DSSC, the commonly used organic dyes are the N719 sensitizing dye for TiO_2 material and the D205 organic dye for ZnO material (Diguna et al., 2020). The potential in this research is that AgNPs are composited into the organic dye D205 with variations of 0 wt% and 10 wt%, which are expected to enhance the light absorption and electron transfer processes of the organic dye D205 because AgNPs can increase light absorption in the visible region due to their advantage in the optical phenomenon of Localized Surface Plasmon Resonance (LSPR) (Kolya et al., 2019). AgNPs enhance light absorption in solar cells through surface plasmon resonance, increasing the device's light trapping capacity and resulting in superior photovoltaic properties (Kure et al., 2024).

AgNPs themselves were synthesized using the Green Synthesis method by reducing Moringa Oleifera extract with $AgNO_3$ as the reductant. The use of Moringa Oleifera extract has various advantages, namely being environmentally friendly, easily accessible, and relatively low-cost (Fitriany et al., 2023). Moringa Oleifera itself has various functions, one of which is polysaccharides as bioactive (Adeyi et al., 2023). The structure and crystalline properties of the synthesized AgNPs were confirmed by XRD, with four main peaks in the XRD spectrum showing distinct peaks at $2\theta = 38.23°, 44.42°, 64.44°$, and $77.39°$, corresponding to hkl (111), (200), (220), and (311) respectively (Panja et al., 2016). Scanning electron microscopy was used to investigate the morphology and determine the stability of silver nanoparticles, Ca-mellia sinensis, showing AgNPs morphology in the form of spherical clusters with a characteristic diameter of 26.9 nm (Khalid Mohamed et al., 2021).

In previous research, green synthesis AgNPs have been studied in various fields using different plant extract bioreduction, one of which is in the health sector. Green Synthesis AgNPs using Areca Catechu extract as a bioreductor can be used as an antibacterial agent (Azizah, 2022), Green Synthesis AgNPs using Aegle Marmelos leaf extract can be utilized as an anticancer agent (Rama et al., 2023), and Green Synthesis AgNPs using L. acapulcensis extract can be utilized as an antimicrobial coating agent (Garibo et al., 2020). From previous research, not much has been studied regarding the green synthesis of AgNPs using Moringa Oleifera extract as a reductant. Meanwhile, Moringa Oleifera extract is a plant that grows abundantly in Indonesia, making it easy to find. In previous research, metal materials and dyes successfully increased dye absorbance, namely AuNPs composited with Dye N719, which enhanced dye absorbance in the visible region up to the wavelength range of 400-700 nm (Pujiarti et al., 2019). From previous research, AgNPs were utilized as antibacterial and antimicrobial agents. Still, there has been little study on AgNPs to enhance the absorbance of the organic dye D205 until Pujiarti's research demonstrated the success of metal materials in increasing dye absorbance. However, the materials used were not AgNPs and the organic dye D205. The novelty of this research is that AgNPs composited with the organic dye D205 at 0 wt% and 10 wt% variations can enhance the absorbance of the organic dye D205 on ZnO nanorod electrodes synthesized using the hydrothermal method.

RESEARCH METHODS

The method used in this study is the Green Synthesis method with Moringa oelifera extract as the reducing agent for $AgNO_3$ material (Yoga Darmawan et al., 2023). Moringa oelifera leaves were locally obtained from healthy Moringa oelifera trees growing along the banks of the Dam Kali Amprong River in Kedungkandang, Malang City. Moringa oelifera leaves are collected locally, thoroughly cleaned with Deionized water to remove all visible contaminants, then chopped into small pieces and dried at room temperature. $AgNO_3$ and aquades were also used at various stages in this research.

Synthesis of AgNPs

The synthesis of AgNPS, as shown in (Figure 1), begins with dissolving 20 grams of Moringa oelifera extract in 200 ml of distilled water, then stirring for 24 hours at a speed of 400 rpm (Asif et al., 2022). The resulting solution was filtered using filter paper to obtain the Moringa oleifera extract solution. Then, 1 M $AgNO_3$ was dissolved in aquades and stirred for 15 minutes at a speed of 400 rpm at room temperature, after which the $AgNO_3$ solution was mixed with the Moringa Oelifera extract solution, with a ratio of 9:1 $AgNO_3$ to Moringa Oelifera extract. The mixture obtained from the $AgNO_3$ solution and Moringa oleifera extract was then heated under direct sunlight for 15 minutes until the colour of the solution darkened. Next, centrifugation was performed to obtain the precipitate of the AgNPs solution at a speed of 3200 rpm for 5 minutes, and then the resulting AgNPs precipitate was dried using an oven at 80˚C for 24 hours to obtain AgNPs powder (Fabiani et al., 2019).

AgNPs—D205 Composite

The process of AgNPs composite with the organic dye D205, as shown in (Figure 1), is the first step, where AgNPs powder is composited with the organic dye D205 with variations of 0 wt% and 10 wt% AgNPs. The concentration of 10 wt% AgNPs was chosen based on previous studies using an AgSC8 concentration in the N719 dye composite at 8.57 wt%, which showed optimum absorbance (Rahayu et al., 2022). Thus, at a concentration of 10 wt%, the plasmonic effect of AgNPs is at an optimal intensity to enhance the absorbance of organic dyes. Meanwhile, 0 wt% is used as a control to evaluate the specific influence of the presence of AgNPs on the performance of the ZnO nanorod electrode. The concentration of the organic dye D205 is 1 mM, which is 0.008 grams of the organic dye D205 composited with 0.0008 AgNPs dissolved in acetonitrile and stirred for 3 hours at a speed of 500 rpm. Next, the ZnO nanorod electrode was immersed in the D205 organic dye composite AgNPs solution for 24 hours.

Characterization

The AgNPs powder produced from green synthesis with Moringa oleifera extract was analyzed to determine the crystal structure and identify the phases of the formed material using an X-ray diffractometer, specifically XRD (Shimadzu XD-3H) with Cu-Kα radiation (λ = 1.5406 Å) in continuous scanning mode $2\theta = 30^\circ - 80^\circ$ (Maghfury & Anggono, 2020). AgNPs powder was also characterized using a Scanning Electron Microscope (SEM), which is a microscope with high magnification of up to 20,000x to visualize the sample morphology using scanning and electron emission (Sahdiah & Kurniawan, 2023). Thin film ZnO nanorod electrodes were immersed in a D205 organic dye solution with AgNPs composites, with variations of 0% and 10% AgNPs, and analyzed to determine the absorption band spectroscopy and absorbance using UV-Vis testing on the Analytik Jena Specord 200 plus.

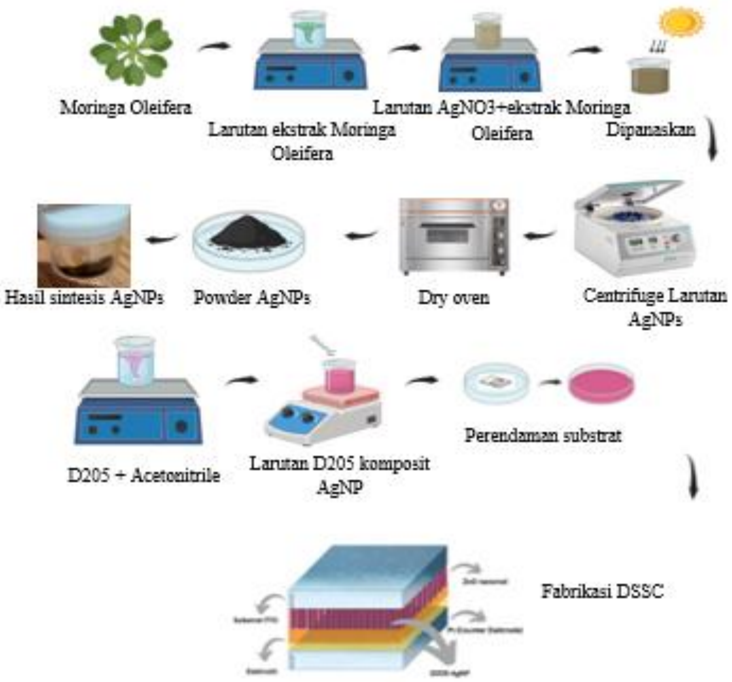

Figure 1. AgNPs synthesis pipeline

RESULTS AND DISCUSSION

The XRD analysis results of AgNPs show several Bragg phases that match the reference and were refined using the Rietica software with the AMCSD-No. 0015127 database. Reflection peaks were produced at 2θ values of 37.98°, 44.1°, 64.41°, and 77.48°, indexed as (111), (200), (220), and (311), with other phases identified as impurities as referenced in (Abdel-Rahman et al., 2022). The structure of AgNPs based on their crystal structure is Face-Centered Cubic (FCC) (Haris & Ahmad, 2024). The XRD results clearly show that the AgNPs synthesized with Moringa Oleifera extract have a natural crystal form with several impurity peaks indicating the presence of organic materials associated with Moringa oleifera (Haris & Ahmad, 2024). The crystal size of AgNPs was calculated using the Scherrer equation, namely $D=k\lambda/(\beta \cos \theta)$, where D is the crystal size (nm), λ is the wavelength of Cu Kα X-ray (0.15406 nm), k is the Scherrer constant (0.9), β is the FWHM (rad), and θ is the Bragg angle (Aouf et al., 2024).

Table 1. Refiment XRD

	Crystal size (nm)	*Rp* (%)	*Rwp* (%)	χ^2	Kisi Kristal	*a*	*b*	*c*	density
Ag Model					F m 3 m	4.058	4.058	4.058	10.722
AgNP	34	15.55	20.90	2.942	F m 3 m	4.0866	4.0866	4.0866	10.492

The crystal size of AgNPs obtained in this study is listed in (Table 1) as 34 nm, while in the previous study, the crystal size was around 23 nm (Muhammad et al., 2023). The results of the analysis using Rietica software show relatively low Rp and Rwp values, indicating a good crystal phase. However, the presence of peaks higher than the main peak indicates a high level of sample impurity.

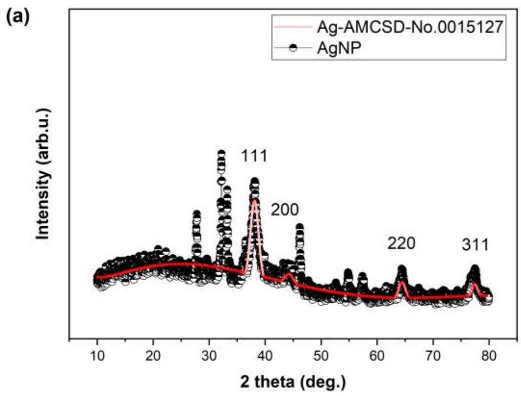

Figure 2. Diffractogram of AgNPs Powder

The results of the SEM-EDX analysis of AgNPs shown in (Figure 3) indicate the morphology of AgNPs powder, with a uniform morphology of AgNPs and an average diameter size as shown in (Table 2), with an ideal particle size of 50 nm-60 nm (Sathishkumar et al., 2020).

Table 2. The diameter size and porosity of AgNPs powder

Material	Diameter (nm)	Height (nm)	Porosity (%)
AgNP	85.125	-	57.15

Most AgNPs are spherical or nearly spherical, as seen in previous research references with a particle size of 26.9 nm forming spherical clusters, while some others are irregular or polygonal in shape, indicating that the AgNPs powder sample is stable and does not show nanoparticle agglomeration (Khalid Mohamed et al., 2021).

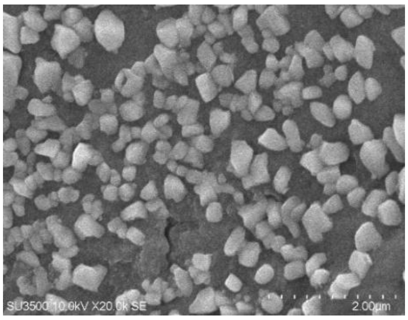

Figure 3. SEM *powder* AgNPs

The porosity of AgNPs was determined using the Origin software, yielding a value of 57.15%. The atomic composition of AgNPs material from SEM-EDX (Energy Dispersive X-Ray) tests to explain the chemical elements contained in the sample and the estimated atomic ratios is shown in (Figure 4) with the atomic ratios written in (Table 3).

Table 3. Composition of Atom powder AgNPs

Material	Atom	Weight (%)	Atom (%)
AgNPs	AgL	79.64	37.35
	C K	5.70	24.71
	O	5.07	16.02
	N K	3.52	12.71
	F K	0.45	1.19
	Na K	0.09	0.21
	Mg K	0.09	0.18
	Si K	0.32	0.57
	P K	1.45	2.36
	S K	1.11	1.75
	Cl K	2.40	3.43
	Ca K	0.16	0.21

The EDX spectrum of AgNP shows high intensity with an Ag ratio of 79.64%, confirming the successful formation of AgNPs (Figure 4).

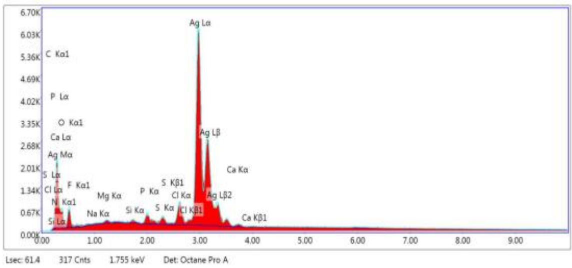

Figure 4. SEM -EDX *powder* AgNP

Other contained elements originate from the biomolecules of Moringa oleifera that adhere to the surface of AgNP, thereby confirming the participation of plant biomolecules as stabilizers of the nanostructure (Adeyi et al., 2023).

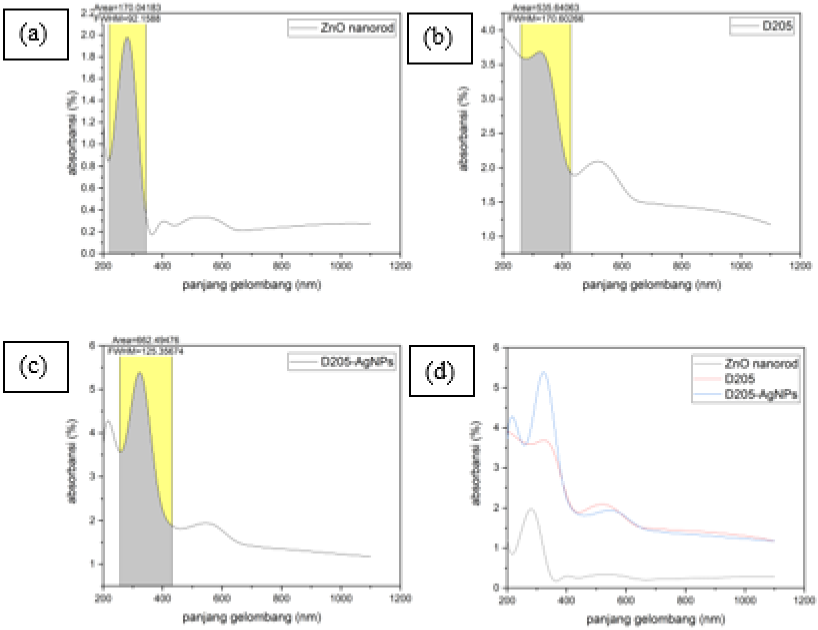

Figure 5. Absorbansi (a) elektroda ZnO nanorod (b) dye Organik D205 (c) D205-AgNPs (d) samples a-c

UV-Vis spectroscopy is one of the techniques used to determine the band gap in the absorption region and the absorbance of AgNPs. In (Figure 5) (Table 4), the absorbance data show that absorbance increases with the addition of the organic dye D205 (0 wt%) and the composite of the organic dye D205 with AgNPs (10 wt%) on the ZnO nanorod electrode.

Table 4. Absorbance Data

No.	Sampel	Wavelength (nm)	Absorption area
1.	ZnO nanorod	283	170.04
2.	D205	321	535.64
3.	D205-AgNPs	325	662.49

This is due to the properties of AgNPs, specifically LSPR, which is indicated by the decrease in the absorption peak in the particle geometry. This can affect the plasmon resonance frequency, thereby increasing the light absorption of the organic dye D205. In the ZnO nanorod electrode sample, absorbance was obtained at a wavelength of 283 nm, with the highest absorption area at 170.04 nm. In the ZnO nanorod electrode sample that has been immersed in the organic dye D205, the absorbance is at a wavelength of 321 nm with an absorption area of 535.64 nm. Meanwhile, in the ZnO nanorod electrode sample that has been immersed in a composite of organic dye D205 and 10 wt% AgNPs, the absorbance is at a wavelength of 325 nm with an absorption area of 662.49 nm. This indicates the success of this research, namely that AgNPs synthesized using the Green Synthesis method with Moringa Oelifera extract can enhance the absorbance of the organic dye D205 on the ZnO nanorod electrode. AgNPs that have been composited with the organic dye D205 on the ZnO nanorod electrode show a decrease in the bandgap, as seen in (Table 5). This occurs due to the influence of optical phenomena present in AgNPs, which can enhance light absorption more effectively in the visible light region. From this study, the band gap is shown in (Figure 6). On the ZnO nanorod electrode, a band gap of 3.06 eV and 3.78 eV was obtained (Siagian et al., 2022). The bands in the sample soaked in the organic dye D205 were obtained at 3.69 eV, and in the composite of the organic dye D205 with AgNPs, the band gap was obtained at 2.99 eV. In previous research, a decrease in the band gap resulted in an increase in DSSC efficiency from 1.58% to 2.04% (Rahayu et al., 2022).

Table 5. Band gap Data

No.	Sample	Band Gap I (eV)	Band Gap II (eV)
1.	ZnO nanorod	3.06	3.78
2.	D205	3.69	
3.	D205-AgNPs	2.99	3.88

The results of the increased absorption indicate that AgNPs are capable of enhancing light absorption through LSPR plasmon resonance, enabling effective energy localization. The three-layer structure proposed in this paper uses AgNPs to achieve precise LSPR regulation, enhancing its application in nanotechnology and SERS substrates (Lin et al., 2024). Thus, the implications of this research can contribute to improving the results of DSSC research and the performance of DSSC in future studies.

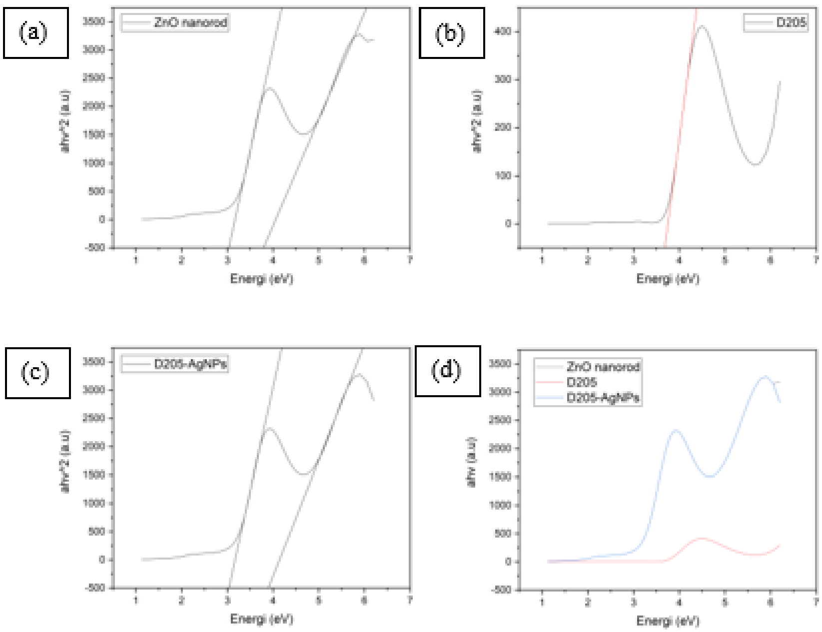

Figure 6. Band gap (a) elektroda ZnO nanorod (b) dye Organik D205 (c) D205-AgNPs (d) samples a-c

CONCLUSION

This research has successfully synthesized AgNPs using the green synthesis method with Moringa Oleifera extract. In the XRD results, the AgNPs powder was confirmed using Rietica software, showing Ag peaks with the main peak at hkl 111. The SEM-EDX results show that AgNPs have been formed with an average particle size of 85 nm. AgNPs themselves have successfully increased the absorbance of the organic dye D205 on the ZnO nanorod electrode, as evidenced by the UV-Vis absorbance data analysis, which shows an increase when AgNPs are composited with the organic dye, specifically at the 0 wt% and 10 wt% variations being 321 nm and 325 nm. The band gap produced also shows a decrease in the band gap at variations of 0 wt% and 10 wt%, which are 3.69 eV and 2.99 eV, respectively. This is the influence of the properties of AgNP, particularly LSPR, which is indicated by the decrease in the absorption peak in the particle geometry. This can affect the plasmon resonance frequency, thereby increasing the light absorption of the organic dye D205.

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