

The Effect of Varying Concentrations of Secang Wood Extract (*Biancaea sappan* L.) on the Characteristics of TiO₂ Produced by Green Synthesis

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Abstract

Green synthesis using plant-derived extracts offers an alternative pathway for modifying the structural and optical properties of titanium dioxide (TiO₂). However, the effects of varying volumes of sappan wood (*Biancaea sappan* L.) extract on these properties, compared with conventional synthesis, require further investigation. This study investigates the effect of adding sappanwood extract on the characteristics of TiO₂, compared with conventional synthesis without the extract. Titanium (IV) isopropoxide (TTIP) was used as a precursor, with 5, 10, and 15 mL of sappanwood extract added during synthesis. The materials were calcined at 500 °C and characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and UV-Visible diffuse reflectance spectroscopy (UV-Vis DRS). XRD analysis identified the anatase phase in all samples, and average crystallite sizes, calculated using the Scherrer equation, ranged from 15.84 to 19.74 nm. Meanwhile, the optical band gap increased from 3.03 eV for conventional TiO₂ to 3.23 eV, 3.24 eV, and 3.25 eV for samples prepared with the addition of 5, 10, and 15 mL of extract, respectively. The results reveal a non-monotonic variation in crystallite size and an increase in the optical band gap following the addition of the extract, with only small changes among the extract-assisted samples. These findings establish a relationship between the volume of sappan wood extract and the structural and optical characteristics of TiO₂, providing a basis for further investigation into plant-assisted modification of TiO₂ properties.

Keywords: Green synthesis; titanium dioxide (TiO₂); sappan wood (*Biancaea sappan* L.); crystallite size; optical band gap.

Introduction

Titanium dioxide (TiO₂) is widely used in various industrial and technological applications because of its chemical stability, high refractive index, and semiconductor properties^[1]. Its crystalline phase, crystallite size, and optical band gap are important physicochemical characteristics that can influence its performance in different applications. Conventional TiO₂ synthesis methods include chemical vapor deposition, electrochemical deposition, sol-gel processing,

hydrothermal treatment, and chemical precipitation. Depending on the method and operating conditions, these processes may require substantial energy input or chemical reagents with environmental implications. Green synthesis using plant extracts has therefore attracted interest as an alternative approach that incorporates renewable biological resources into material preparation^{[2], [3]}.

Plant extracts contain diverse phytochemicals, including phenolic compounds and flavonoids, with functional groups that may interact with

precursor species during material synthesis. In the formation of TiO_2 from titanium alkoxide precursors such as titanium (IV) isopropoxide (TTIP), hydrolysis and condensation are the principal chemical processes. Consequently, the role of plant-derived compounds should not be attributed exclusively to the reduction of metal ions. Instead, these compounds may influence precursor interactions, nucleation, crystal growth, and surface characteristics, depending on their composition and the synthesis conditions. Such effects may be reflected in the resulting crystallite size and optical properties, although the specific mechanisms require appropriate experimental evidence ^[4].

Sappan wood (*Biancaea sappan* L.) is a potential source of phytochemicals, including flavonoids and other phenolic compounds. Previous research has investigated the use of sappan wood extract in green TiO_2 synthesis ^[5]. This earlier work provides a basis for further investigation of the synthesis process. However, the effects of varying extract volume on the structural and optical characteristics of TiO_2 need to be evaluated within the specific synthesis conditions employed in the present study. In particular, examining crystallite size and the optical band gap together, using conventionally synthesized TiO_2 as a reference, can help clarify how these two properties vary with the addition of extract. This comparison is relevant because changes in crystallite size do not necessarily correspond directly to changes in optical band gap.

Accordingly, the present study extends previous work on sappan wood extract-assisted TiO_2 synthesis by investigating the effects of adding 5, 10, and 15 mL of extract to the synthesis system, with TiO_2 prepared without extract serving as a conventional reference. The materials were calcined at 500 °C and characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD),

and UV-visible diffuse reflectance spectroscopy (UV-Vis DRS). FTIR was used to examine functional groups, XRD to identify crystalline phases and estimate average crystallite sizes using the Scherrer equation, and UV-Vis DRS to determine the optical band gap. By comparing the structural and optical characteristics of the resulting materials, this study provides an experimental basis for assessing how the volume of sappan wood extract affects TiO_2 properties under the synthesis conditions investigated.

Experimental

Materials

The materials used in this study were distilled water, ethanol (96% for extraction; Merck), commercial sappan wood (*Biancaea sappan* L.) obtained from Pontianak, West Kalimantan, titanium(IV) isopropoxide (TTIP, 97%; Sigma-Aldrich), and nitric acid (HNO_3 , 65%; Merck). Analytical-grade ethanol (Pro Analysis, p.a.) was used for the synthesis procedures.

Equipment

The equipment used in this study included a stirring rod, petri dish, funnel, desiccator, beaker glass, measuring cylinder, hot plate, filter paper, erlenmeyer flask, volumetric flask, fume hood, magnetic stirrer, mortar and pestle, oven, dropper pipette, graduated pipette, rotary evaporator, spatula, and analytical balance. The analytical instruments employed were a Fourier transform infrared (FTIR) spectrophotometer (Shimadzu IRPrestige-21), an x-ray diffraction (XRD) instrument (Bruker D2-Phaser), and a diffuse reflectance spectroscopy (UV-Vis DRS) instrument (Perkin Elmer 365+).

Method

Extraction of sappan wood (Biancaea sappan L.)

The Sappan wood obtained from Pontianak, West Kalimantan, was extracted following a previously reported procedure ^{[6], [7]}. A total of 1 kg of sappan wood chips was macerated in 10 L

of 96% ethanol for three consecutive 24 h periods at room temperature, with stirring every 24 h. The macerate was filtered, and the filtrate was concentrated using a rotary evaporator at 60 °C and 120 rpm. The concentrated extract was stored in a tightly closed, dark-colored bottle until use. The extraction yield was not determined.

Before synthesis, the concentrated sappan wood extract was diluted with ethanol to a final volume of 30 mL. Aliquots of 5, 10, and 15 mL of this diluted extract were used in the respective TiO₂ synthesis experiments.

Green Synthesis of TiO₂

The synthesis procedure was adapted from a previously reported method^[8], with modifications to examine the effects of varying volumes of sappan wood extract. Briefly, 25 mL of ethanol was placed in a beaker, followed by the addition of 5 mL of TTIP (97%). Subsequently, 5, 10, or 15 mL of the diluted sappan wood extract was added to the mixture. The mixture was heated at 50 °C and stirred for 2 h using a magnetic stirrer. The stirring speed was not recorded. After the reaction, the resulting solid was separated by filtration and dried at room temperature for 24 h. The dried material was calcined at 500 °C for 2 h using a heating rate of 5 °C min⁻¹. The calcined product was ground into a fine powder and stored in tightly sealed containers until characterization by FTIR, XRD, and UV-Vis DRS. The extract volumes of 5, 10, and 15 mL were selected as comparative experimental conditions to evaluate their association with the structural and optical properties of TiO₂. These volumes were not established as optimized conditions before the experiments.

Conventional (Chemical) Synthesis of TiO₂

The conventional synthesis procedure was adapted from a previously reported method^[9].

Briefly, 20 mL of distilled water was mixed with 5 mL of ethanol, then 5 mL of TTIP was added. The mixture was stirred for 1 h. Nitric acid (HNO₃, 65%; Merck) was then added until the pH reached 2. The volume of HNO₃ required to reach this pH was not recorded. The mixture was stirred for an additional hour and filtered to collect the solid product. The resulting residue was dried at 80 °C for 16 h and calcined at 500 °C for 2 h using a heating rate of 5 °C min⁻¹. The calcined material was ground into a fine powder and stored in tightly sealed containers until characterization by FTIR, XRD, and UV-Vis DRS.

Result and Discussions

Synthesis of TiO₂

TiO₂ was synthesized using both a conventional method and an extract assisted method to investigate the relationship between the volume of sappanwood extract and the structural and optical properties of the resulting material. Plant extracts contain various phytochemicals, including phenolic compounds and other oxygen-containing constituents, which can interact with precursor species or particle surfaces during material synthesis^[10]. In this study, the extract was added as a potential stabilizing agent or surface modifying agents during TiO₂ synthesis^[11].

The formation of titanium dioxide from titanium(IV) tetraisopropoxide (TTIP) primarily involves hydrolysis and condensation reactions. The hydrolysis of titanium alkoxide groups in the presence of water generates titanium species containing hydroxyl groups, which subsequently undergo condensation to form Ti-O-Ti bonds. These processes contribute to the formation of an inorganic oxide network. The addition of plant extract may influence the local chemical environment or the subsequent growth of the oxide material. However, the specific interactions between individual phytochemicals

and the titanium containing species were not directly determined in this study^[12].

Figure 1 shows TiO₂ powders synthesized via the conventional method and three different extract assisted conditions. The conventional sample yielded 39.94%, while samples prepared with the addition of 5, 10, and 15 mL of extract yielded 26.35%, 26.74%, and 27.05%, respectively. Thus, the extract assisted samples exhibited lower yields than the conventional sample under the conditions used. The yield increased slightly as the extract volume rose from 5 to 15 mL, although the change was relatively minor.

These differences in yield reflect the quantity of product obtained, but they do not, by themselves, indicate differences in reaction conversion or synthesis efficiency. Such a conclusion requires additional information on product composition, recovery losses, and the conversion rate of the precursors. Therefore, the influence of the extract volume was further evaluated using FTIR, XRD, and UV-Vis DRS techniques.

FTIR Analysis

FTIR spectroscopy was used to investigate the functional groups present in the sappan wood extract and the synthesized TiO₂ samples. Figure 2 shows the spectra recorded over the wavenumber range of 400–4000 cm⁻¹. The extract exhibited a broad absorption band at approximately 3394.72 cm⁻¹, attributed to O-H

stretching vibrations. This feature is consistent with the presence of hydroxyl-containing constituents, including phenolic compounds, in the extract.

Additional bands at 2935.66 and 2879.72 cm⁻¹ may be assigned to aliphatic C-H stretching vibrations. The absorption band at 1708.93 cm⁻¹ is consistent with C=O stretching, while the band at 1614.42 cm⁻¹ may be associated with aromatic C=C vibrations and potentially overlapping contributions from other oxygen-containing functional groups. The bands at 1033.85 and 877.61 cm⁻¹ may be attributed to C-O-C stretching and aromatic C-H bending vibrations, respectively. These assignments indicate the presence of several functional groups but do not uniquely identify individual phytochemicals.

The spectra of the synthesized samples exhibited absorption bands in the lower-wavenumber region at 582.50 cm⁻¹ for TiO₂-C, 545.85 cm⁻¹ for TiO₂-5 mL, 588.29 cm⁻¹ for TiO₂-10 mL, and 590.22 cm⁻¹ for TiO₂-15 mL. These bands are consistent with Ti-O-related lattice vibrations, including Ti-O-Ti vibrations, and support the formation of titanium oxide materials. The spectra also showed absorption features in the O-H region and near 1600–1650 cm⁻¹. These features may be associated with surface hydroxyl groups, adsorbed water, or overlapping vibrations; therefore, they cannot be attributed exclusively to residual plant-derived compounds^[13].



Figure 1. Synthesized TiO₂ a) TiO₂-C, b) TiO₂-5mL, c) TiO₂-10mL, d) TiO₂-15mL

Differences between the extract spectrum and the spectra of the synthesized materials suggest changes in the chemical environment of the functional groups during synthesis and subsequent thermal treatment. Nevertheless, the observed spectral differences do not directly demonstrate chemical bonding between a specific phytochemical and the TiO_2 surface. Similarly, changes in band intensity alone cannot establish a reduction mechanism, particularly because quantitative intensity ratios, replicate spectra, and complementary surface-sensitive characterization were not available [14].

The FTIR results therefore provide evidence of characteristic functional groups in the extract and Ti-O related vibrations in the synthesized materials. The possible contribution of sappan wood phytochemicals to TiO_2 formation remains a plausible interpretation, but the precise molecular interactions and their

influence on nucleation and crystal growth require further investigation [15].

X-Ray Diffraction Analysis and Crystallite Size

Figure 3 presents the XRD patterns of the conventionally synthesized and extract-assisted TiO_2 samples recorded over a 2θ range of 20° – 80° . The diffraction patterns were compared with the reference data reported in the original manuscript (JCPDS File No. 01-078-2486). The extract-assisted samples exhibited characteristic reflections at approximately $2\theta = 25.35^\circ$, 37.8° , 48.1° , 53.9° , 55.1° , and 62.7° , corresponding to the (101), (004), (200), (105), (211), and (204) planes of anatase TiO_2 , respectively. These reflections indicate that anatase was the principal crystalline phase identified in the extract-assisted samples [16].

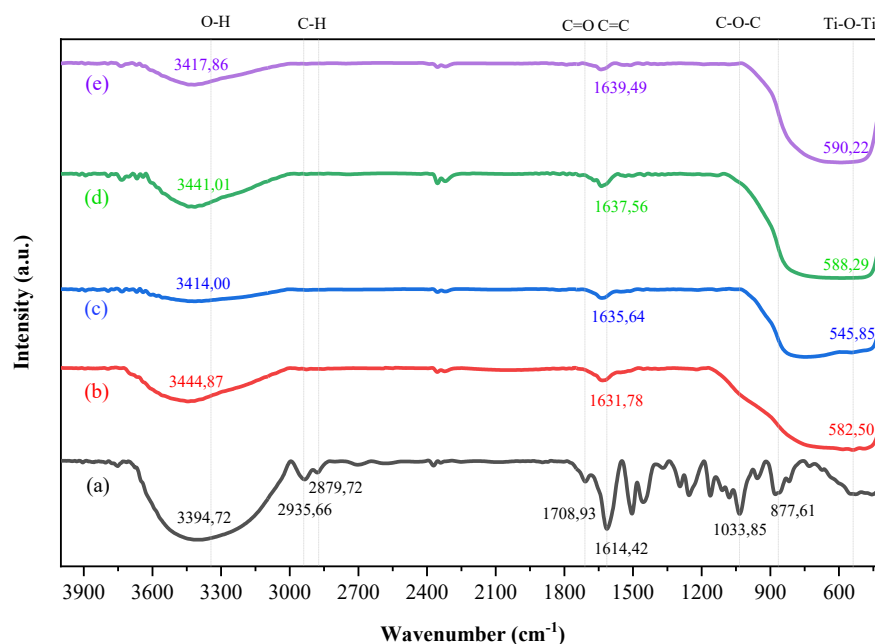


Figure 2. FTIR spectra of a) Sappan wood extract (*Biancaea sappan* L.), b) TiO_2 -C, c) TiO_2 -5mL, d) TiO_2 -10mL, and e) TiO_2 -15mL

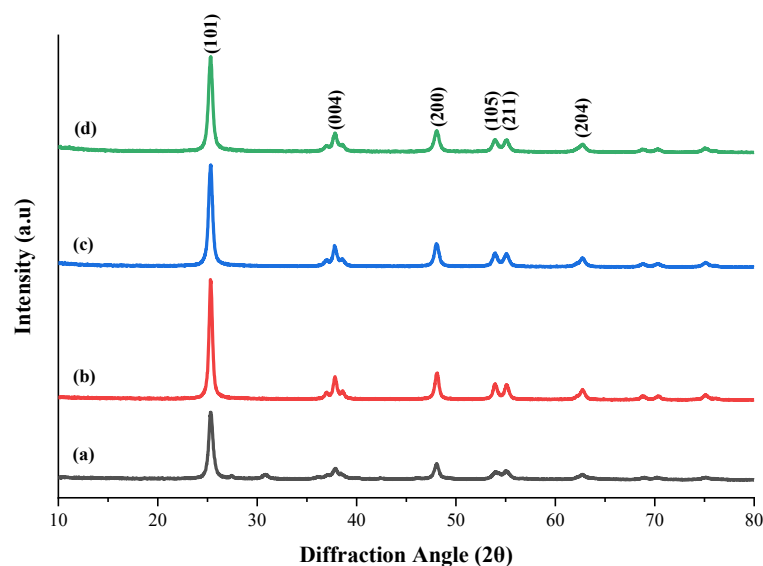


Figure 3. XRD patterns of a) $\text{TiO}_2\text{-C}$, b) $\text{TiO}_2\text{-5mL}$, c) $\text{TiO}_2\text{-10mL}$, and d) $\text{TiO}_2\text{-15mL}$

The conventional sample, $\text{TiO}_2\text{-C}$, exhibited an additional reflection at approximately $2\theta = 27.413^\circ$, consistent with the (110) reflection of rutile TiO_2 . This observation suggests the presence of a rutile component alongside anatase in the conventional sample. No comparable rutile reflection was identified in the reported patterns of the extract-assisted samples. However, quantitative phase refinement was not performed; consequently, the relative proportions of anatase and rutile cannot be determined from the available data.

The observed difference in phase composition may be associated with differences in the chemical environment and reaction conditions during synthesis. In the conventional method, nitric acid was used to adjust the mixture to pH 2, whereas the extract-assisted method involved adding sappan wood extract. These differences provide possible chemical factors that could influence hydrolysis, condensation, and subsequent crystallization. Nevertheless, the present data do not establish which factor, if any, caused rutile formation in $\text{TiO}_2\text{-C}$. A more definitive explanation would require controlled comparisons and quantitative phase analysis^[17].

The average crystallite sizes were calculated using the Scherrer equation based on the full width at half maximum (FWHM) of the selected diffraction peaks. The calculated average crystallite sizes were 15.84 nm for $\text{TiO}_2\text{-C}$, 19.75 nm for $\text{TiO}_2\text{-5 mL}$, 18.50 nm for $\text{TiO}_2\text{-10 mL}$, and 16.58 nm for $\text{TiO}_2\text{-15 mL}$.

The extract assisted samples exhibited a non-monotonic trend in calculated crystallite size. The value increased from 15.84 nm for the conventional sample to 19.75 nm for the sample prepared with 5 mL of extract, then decreased to 18.50 and 16.58 nm as the extract volume increased to 10 and 15 mL, respectively. Thus, the highest calculated crystallite size was obtained at 5 mL of extract, whereas the value at 15 mL approached that of the conventional sample.

One possible interpretation is that changes in extract volume may alter the chemical environment of titanium-containing species or particle surfaces, thereby influencing the development of crystalline domains. However, the hydrolysis, condensation, and crystal-growth processes were not monitored directly, so this interpretation should not be considered a demonstrated mechanism. Furthermore, the

Scherrer equation estimates the size of coherently diffracting crystalline domains rather than the overall particle size. The calculated values, therefore, cannot independently establish particle morphology, agglomeration, or particle-size distribution. Overall, the XRD results indicate that the extract-assisted samples were predominantly identified with the anatase phase and exhibited calculated crystallite sizes in the nanometer range. The variation in crystallite size across the investigated extract volumes was not linear. Further quantitative phase analysis and complementary structural characterization would be needed to determine phase fractions and clarify the origin of the observed differences.

Diffuse Reflectance Spectroscopy (UV-Vis DRS)

UV-Vis diffuse reflectance spectroscopy was used to investigate the optical properties of the synthesized TiO₂ samples [18]. The reflectance data were processed using the Kubelka–Munk function, and the optical band gap values were estimated by extrapolating the linear regions of the Tauc plots to the energy axis, as shown in

Figure 4. The estimated band gap values were 3.03 eV for TiO₂-C, 3.23 eV for TiO₂-5 mL, 3.24 eV for TiO₂-10 mL, and 3.25 eV for TiO₂-15 mL.

The extract-assisted samples exhibited higher estimated band gap values than the conventional sample, with differences of 0.20–0.22 eV. In contrast, the differences among the three extract-assisted samples were small, ranging from 0.01 to 0.02 eV. These results indicate that the principal observed difference was between the conventional and extract-assisted samples, whereas increasing the extract volume from 5 to 15 mL was associated with only a modest change in the estimated optical band gap.

The lower band gap estimated for TiO₂-C may be related to its different XRD pattern, which included a reflection consistent with rutile TiO₂ in addition to the anatase reflections. Differences in phase composition can influence the optical response of TiO₂. However, the available results do not establish that the rutile component alone accounts for the lower band gap, particularly since quantitative phase fractions were not determined.

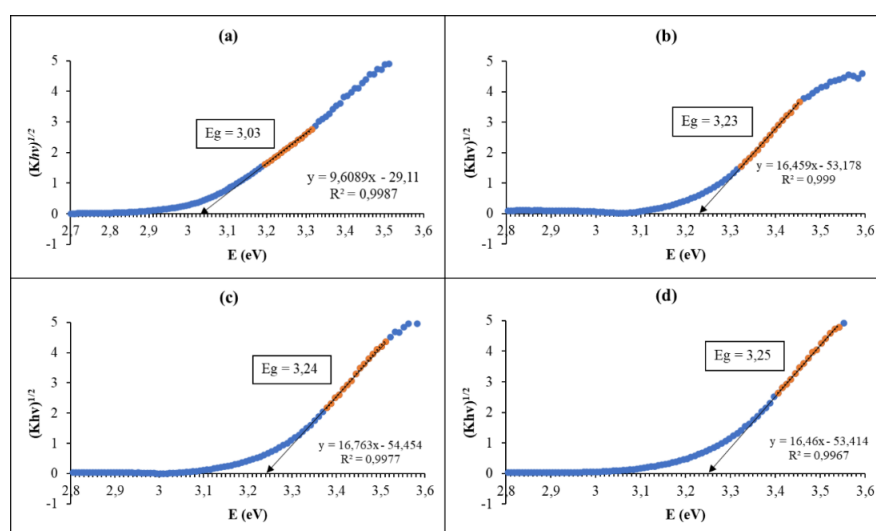


Figure 4. Band Gap Energy Curves a) TiO₂-C b) TiO₂-5mL c) TiO₂-10mL d) TiO₂-15mL

Other factors, including crystallinity, defect-related electronic states, oxygen vacancies, surface chemistry, and residual organic species, may also affect the optical response. These factors were not independently characterized in this study and therefore remain possible explanations rather than confirmed causes. In addition, the slightly higher band gap values observed in the extract-assisted samples cannot be attributed solely to the drying process, as the drying temperature was not independently varied while the other synthesis conditions were held constant [19].

The small differences among the estimated band gap values of 3.23, 3.24, and 3.25 eV should also be interpreted with caution, as replicate measurements and uncertainty estimates were not available to determine whether these differences exceed experimental variation. Accordingly, the results support a descriptive comparison of the optical band gaps under the investigated synthesis conditions but do not establish a statistically significant effect of extract volume [20].

In summary, the UV-Vis DRS results reveal a difference in the estimated optical band gap between conventional and extract-assisted TiO₂, together with relatively small variations among the extract-assisted samples. Further studies incorporating quantitative phase analysis, complementary defect and surface characterization, and direct photocatalytic testing would be required to clarify the relationship between synthesis conditions, material structure, optical properties, and photocatalytic performance.

Conclusions

This study comparatively evaluated the effects of adding 5, 10, and 15 mL of sappan wood (*Biancaea sappan* L.) extract on the structural and optical properties of TiO₂ relative to conventional synthesis. The crystallite size

exhibited a non-monotonic trend, increasing from 15.84 nm for conventional TiO₂ to 19.75 nm with 5 mL of extract, then decreasing to 16.58 nm with 15 mL. Meanwhile, the estimated band gap increased from 3.03 eV to 3.23–3.25 eV in the extract-assisted samples. These findings suggest that the addition of the extract is associated with changes in TiO₂ crystallite size and optical properties, potentially through interactions that influence material formation; however, the underlying mechanisms remain unverified. TiO₂ formation primarily involves hydrolysis and condensation rather than reduction of Ti⁴⁺ to metallic titanium. Further studies involving quantitative phase analysis, surface characterization, and direct photocatalytic testing are needed to clarify the structure–property relationships and evaluate the potential of these materials for environmental remediation.

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Declaration of Generative AI and AI-assisted Technologies

ChatGPT (OpenAI) and Grammarly were used to improve the readability, grammar and language clarity of the manuscript. These tools were used solely for language editing and did not replace the interpretation of the research findings. The Authors take full responsibility for the accuracy, originality, citations, confidentiality, and final content of the manuscript.

Author Contributions

Conceptualization, R.S.; methodology, R.S.; validation, B.B.; formal analysis, V.; data

curation, A.; writing—original draft, V. and R.S.; writing—review and editing, W.R. and A.

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Declaration of Conflicts of Interest

The authors declare no conflict of interest.

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