

Mekanika: Majalah Ilmiah Mekanika

Mechanical Properties of Unsaturated Polyester Composites Reinforced with Eggshell-Derived CaCO_3 Bio-filler as a Sustainable Material

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Abstract

The increasing demand for sustainable engineering materials has encouraged the development of polymer composites reinforced with bio-based fillers. Eggshell waste, which contains more than 90% biogenic calcium carbonate, represents an abundant and low-cost resource with potential as a reinforcing agent in thermoset composites. In this study, unsaturated polyester resin Bayesian Quantitative Trait Nucleotide (BQTN) 157 was reinforced with eggshell-derived calcium carbonate particles sized 105-149 μm , incorporated at volume fractions of 1-5%. Test specimens were produced using standardized moulds and evaluated for tensile properties in accordance with American Society for Testing and Materials (ASTM) D638 and for impact resistance using the Izod in ASTM D4812. The tensile results indicated a reduction in Ultimate Tensile Strength (UTS) of 6.7%, from 20.85 MPa for neat resin to 19.46 MPa at 5% filler, while the elastic modulus increased by 9.7%, from 1.44 GPa to 1.58 GPa. The impact resistance improved with filler addition, reaching a maximum of 91.75 J/m at a 3% filler concentration, representing a 15.7% increase, before slightly declining at higher concentrations. These findings demonstrate that eggshell-derived calcium carbonate is a promising sustainable bio-filler, with 3% volume identified as the optimum composition for practical composite applications.

1 Introduction

Material technology evolves in response to the demand for engineering materials that are not only strong and durable but also environmentally friendly and cost-effective. One promising approach is to use bio-based fillers sourced from waste materials to partially replace synthetic fillers in polymer composites. Synthetic fillers, such as talc or mineral-grade calcium carbonate, are widely used in industry; however, their production is energy-intensive and contributes to environmental burdens [1]. In contrast, eggshell waste, which is abundantly generated from households and food industries, contains more than 90% biogenic calcium carbonate (CaCO_3) along with small amounts of organic matter.

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Utilising eggshells as a composite filler offers a dual benefit: reducing waste disposal problems and decreasing the need for virgin mineral resources, thereby aligning with the principles of the circular economy [2-4]. Previous studies have shown that eggshell-derived CaCO_3 can enhance various mechanical properties in composite materials [5,6]. For instance, Kuila et al. reported increased tensile strength in basalt fibre/epoxy laminates with 5% eggshell CaCO_3 [7,8]. At the same time, Kowshik found that adding similar fillers improved the impact toughness of glass fibre composites. Other works, such as those by Sharaf et al. and Owuamanam et al., demonstrated that natural CaCO_3 in polyester composites can provide performance comparable to synthetic fillers [9,10]. More recent studies have also examined particle size effects, thermal treatment, and surface modification to improve the bonding between filler and matrix [11].

Despite these advances, most studies have been conducted on epoxy or polypropylene matrices, with little attention given to unsaturated polyester particular systems at low filler volume fractions $<5\%$ [5]. The relationship between filler content, stiffness enhancement, and the retention of tensile strength and impact toughness in such systems is still poorly understood [11]. Moreover, limited research has attempted to connect microstructural features, such as particle shape, surface texture, and agglomeration, with the resulting mechanical behaviour. This leaves a gap in understanding how bio-based CaCO_3 fillers perform in polyester systems under controlled processing conditions.

In light of the findings above, the present study investigates the mechanical properties of unsaturated polyester resin BQTN 157 reinforced with thermally treated eggshell-derived CaCO_3 at various volume fractions (1-5%). The novelty of this work lies in its systematic evaluation of tensile strength, elastic modulus, and impact toughness, supported by morphological observations using SEM on the filler, which directly links microstructural features with mechanical performance trends. By identifying the optimum filler content that balances stiffness and toughness, this research contributes scientific insights and practical guidelines for developing low-cost, sustainable composite materials suitable for applications such as automotive panels, lightweight construction boards, and consumer products.

2 Experimental Methods

2.1 Materials

This study used unsaturated polyester resin BQTN 157 produced by PT. Justus Kimiaraya is a composite matrix due to its moderate viscosity, fairly long pot life, and lower cost than technical epoxy resin. The resin has a density of 1.12 g/cm^3 , making it suitable for composite applications where dimensional stability is essential. As a hardener, Methyl Ethyl Ketone Peroxide (MEPOXE), produced by Kawaguchi Kimia Indonesia, was utilized at a resin-to-hardener mass ratio of 100:1. The hardener is a clear liquid initiator with an approximate density of 1.17 g/cm^3 , widely used in polyester curing processes. The natural filler employed was Eggshell Powder (ESP) derived from local chicken eggshells, predominantly composed of CaCO_3 with a density of 2.62 g/cm^3 . The ESP was processed to achieve a particle size in the range of 105-149 μm and incorporated into the composite with volume fractions of 1%, 2%, 3%, 4%, and 5% relative to the resin volume.

2.2. Preparation of CaCO_3

The eggshells were first manually cleaned to remove the adhering membrane, followed by thorough washing under running water to eliminate dust and organic contaminants. Cleaned shells were oven-dried at 80°C for 4 hours to ensure complete removal of residual moisture, as excess moisture can promote agglomeration and interfere with polymer-filler interactions [12]. The dried shells were ground using a high-energy planetary ball mill at 850 rpm for 1 hour to achieve a fine powder, which was subsequently sieved through 100-mesh (150 μm) and 140-mesh (106 μm) sieves. This two-step sieving ensured a controlled particle size distribution in the 105-149 μm range, facilitating adequate dispersion in the polymer matrix [13].

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Subsequently, to improve chemical purity, the sieved eggshell powder was thermally treated by annealing at 500°C for 4 hours in a furnace, allowing decomposition of residual proteins and pigments while preserving CaCO₃ in its calcite form [14,15]. The annealed powders were then gradually cooled to room temperature inside the furnace to avoid thermal shock and stored in airtight containers to prevent moisture uptake before being used in composite preparation [16]. This combination of cleaning, controlled grinding, sieving, and calcination ensured uniform morphology and improved filler stability, essential for reproducible mechanical performance in unsaturated polyester composites [17].

2.3. Composite sample

For each formulation, eggshell powder was weighed according to the volume fraction and slowly added to the BQTN 157 resin. The mixture was then stirred mechanically for 10 minutes to disperse the filler evenly. MEPOXE hardener was added to the resin at a mass ratio of 1%, and then the mixture was stirred slowly for 5 minutes to minimize the formation of air bubbles. The resin-filler mixture was poured into a silicone mold coated with a mold release agent, a dog-bone mold according to ASTM D638 Type I for tensile samples, and a block mold for impact tests. The samples were allowed to harden under laboratory conditions (25 ± 3°C, 50% Relative Humidity (RH)) for 24 hours, then post-cured in an oven at 60 °C for 2 hours until crosslinking. The naming of the sample used the Conformité Européenne (CE) code for eggshell composites, along with a value of 1 to 5 that describes the percentage of volume fraction. Next, the composition of all sample variations is explained in Table 1, which contains the percentage volume of each sample composition [6,18].

Table 1. Variation of test sample composition

Sample Code	Composition		Total Volume (%)	Density (g/cm ³)		Mass (g)		
	Volume Variation							
	Matrix	CaCO ₃		Matrix	CaCO ₃	Matrix	CaCO ₃	Total
CE1	99%	1%	100	1.12	2.62	110.88	2.62	113.5
CE2	98%	2%	100	1.12	2.62	109.76	5.24	115
CE3	97%	3%	100	1.12	2.62	108.64	7.86	116.5
CE4	96%	4%	100	1.12	2.62	107.52	10.48	118
CE5	95%	5%	100	1.12	2.62	106.4	13.1	119.5

2.4. Mechanical testing

Mechanical characterisation included tensile and Izod impact tests, each performed on at least five samples for every filler volume fraction. Tensile tests were conducted using an Instron 3369 universal testing machine (Instron Corp., Norwood, MA, USA) equipped with a 20 kN load cell, at a crosshead speed of 5 mm/min, in accordance with ASTM D638 Type I specimen geometry. The gauge length was set to 50 mm, and strain was measured using an extensometer. Maximum tensile strength and elastic modulus were determined from the stress-strain curves obtained [19]. For impact testing, the unnotched Izod method was applied according to ASTM D4812 [20]. Tests were carried out using a Toyoseiki impact tester (Toyoseiki Co., Tokyo, Japan) with a pendulum mass of 0.4599 kg and an initial pendulum angle of 148°. Unnotched specimens measured 63 ± 0.5 mm in length, 12 ± 0.5 mm in width, and 3.30 ± 0.3 mm in thickness. Impact strength was calculated as the absorbed impact energy divided by the cross-sectional area, and expressed in J/m² [21]. The dimensions of the specimen tested were shown in Figures 1 and 2.

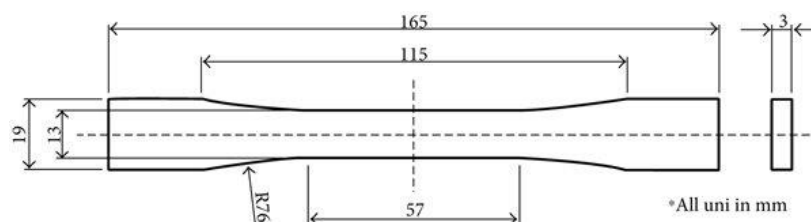


Figure 1. Dimensions of the tensile test sample

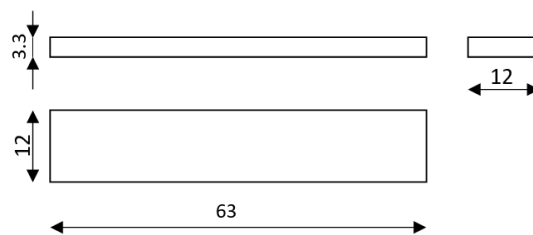


Figure 2. Dimensions of the Izod impact test sample

For clarity, the overall experimental procedure is summarized in a flowchart, as shown in Figure 3. This schematic illustrates the sequential steps followed in material preparation, composite fabrication, and mechanical testing.

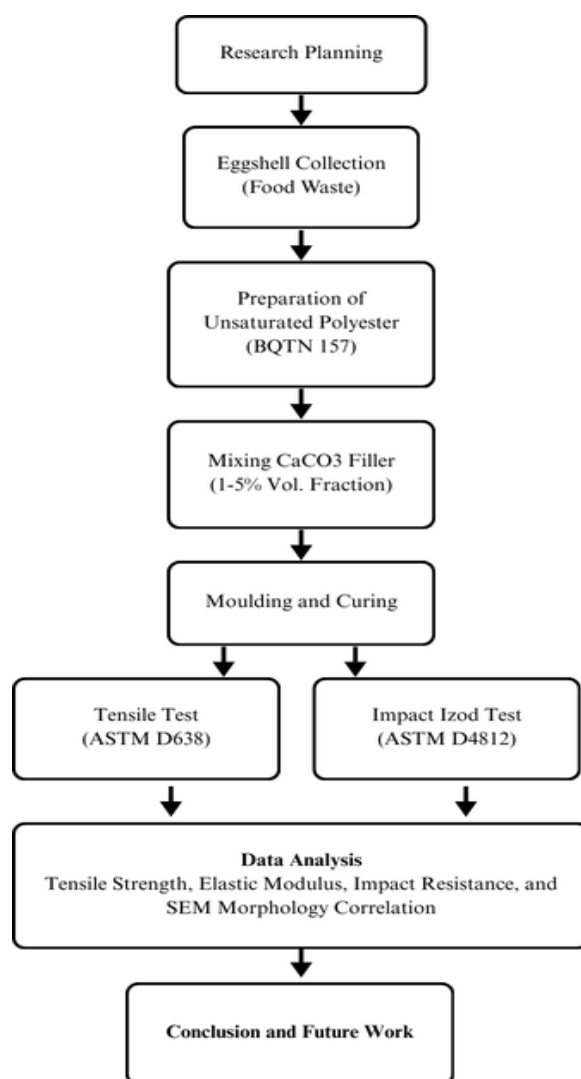


Figure 3. Flowchart of the experimental procedure for unsaturated polyester composites

3 Results and Discussion

3.1. Tensile test result

Tensile tests were conducted to determine the maximum tensile strength and deformation characteristics of the composite at varying CaCO_3 filler volume fractions. Table 2 presents data on the Maximum Force (F), Elongation (Δl), sample dimensions, and Initial Cross-Sectional Area (A_0):

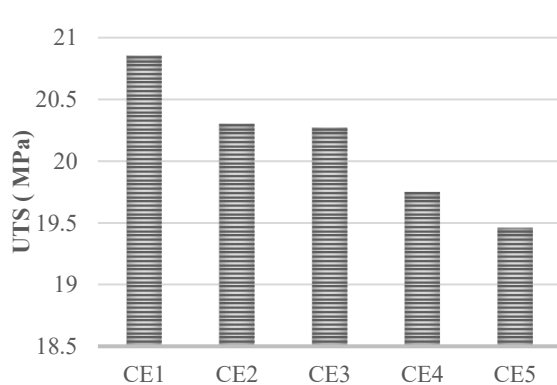
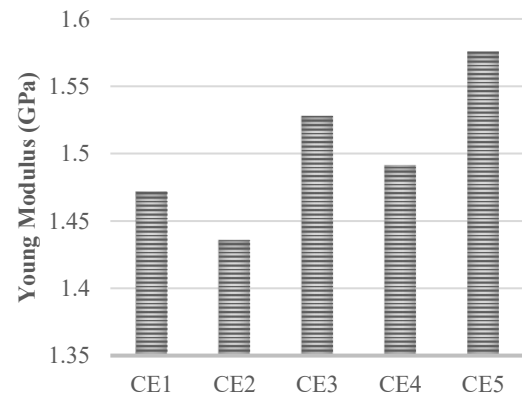
Table 2. Tensile test data

Sample Code	% vf	A_0	F	Δl
		mm ²	N	mm
CE1	1%	47.17	983.61	2.34
CE2	2%	47.41	962.56	2.33
CE3	3%	46.68	946.36	2.19
CE4	4%	47.11	930.36	2.18
CE5	5%	46.97	914.15	2.04

The deformation value Δl also shows a decreasing trend, from 2.34 mm in sample CE1 to 2.04 mm in sample CE5. This decrease indicates that the material becomes stiffer and less elastic as the filler percentage increases. This condition is caused by a less homogeneous distribution of filler material or a weak interface between the filler material and the matrix, thus affecting the material's ability to undergo plastic deformation [1,22].

Figure 4 illustrates the tensile test results of composites with varying volume fractions of eggshell filler, which exhibit a consistent reduction in Ultimate Tensile Strength (UTS) as the filler content increases. The recorded UTS values for samples CE1 through CE5 were 20.85 MPa, 20.30 MPa, 20.27 MPa, 19.75 MPa, and 19.46 MPa, respectively, corresponding to an overall decrease of approximately 6.7% from CE1 to CE5. This decline suggests that higher filler loadings disrupt the continuity of the polymer matrix, weaken intermolecular interactions, and consequently reduce the ability of the composite to withstand tensile stresses [12]. The reduction can be attributed to weak interfacial adhesion between the rigid CaCO_3 particles and the polyester resin, which generates stress concentration zones and accelerates crack initiation under tensile loading. Furthermore, particle agglomeration at elevated filler contents may introduce microstructural defects, limit effective stress transfer, and decrease the matrix's capacity for plastic deformation. These mechanisms indicate that while lower filler levels may provide a slight reinforcement effect, excessive incorporation compromises the tensile performance of the composite [23].

In contrast, the results are presented in Figure 5. reveal an opposite trend in Young's modulus, which gradually increased with higher filler content. Specifically, the modulus improved from 1.44 GPa for CE1 to 1.58 GPa for CE5, with the maximum stiffness observed at the highest filler fraction. This enhancement reflects the intrinsically rigid nature of CaCO_3 , which, when uniformly dispersed within the polymer matrix, restricts molecular chain mobility and increases resistance to elastic deformation [24]. Thus, the eggshell filler acts as a reinforcing phase that enhances stiffness while simultaneously diminishing ductility and tensile strength. This inverse relationship between tensile strength and modulus is characteristic of polymer-filler composites, wherein the incorporation of rigid inorganic particles increases elastic stiffness but reduces fracture resistance. Consequently, optimising the filler volume fraction becomes essential to balance stiffness enhancement and strength retention [25].

**Figure 4.** Ultimate tensile strength**Figure 5.** Young's modulus

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Meanwhile, the elongation parameter showed a decreasing trend with increasing filler fraction, as shown in Figure 6. The elongation value of the sample decreased from 1.42% in CE1 to 1.24% in CE5. This decrease reflects the reduction in material toughness resulting from the addition of filler, which renders the composite structure more fragile or brittle. This is due to the limited movement of the polymer chain, caused by the presence of rigid filler particles, which reduces the material's ability to undergo plastic deformation. Overall, the addition of eggshell filler has a dual effect on the material's mechanical properties. On the one hand, the material becomes stiffer with that increased elastic modulus, but on the other hand, its strength against tensile loads and its ability to undergo plastic deformation decrease [6,26].

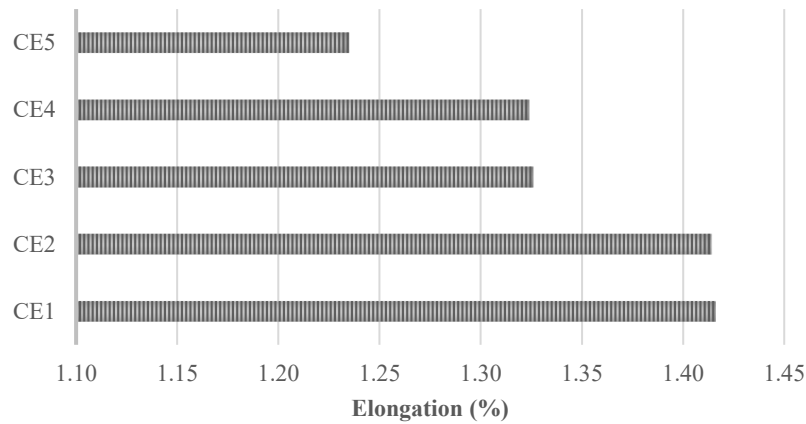


Figure 6. Elongation at break

3.2. Impact test results

The Izod impact test was conducted according to ASTM D4812 on unnotched specimens to assess the resistance of the composites to sudden impact loads. The results are presented in Table 3 and Figure 7, expressed in terms of impact resistance (J/m), which represents the total absorbed energy per unit specimen width. The measured impact resistance values ranged from 31.73 J/m for the composite CE1 with 1% filler to CE3, 36.70 J/m at 3% filler loading. An increasing trend was observed as the filler fraction increased from 1% to 3%, indicating that well-dispersed CaCO_3 particles improved the composite's ability to resist crack initiation and propagation [27]. This enhancement is likely due to crack deflection, matrix deformation, and energy dissipation mechanisms promoted by rigid filler particles [18].

Table 3. The impact test results

Specimen Code	% vf	L (m)	α (°)	β (°)	Thickness (mm)	Width (mm)	Ligament Area (mm ²)	Energy (J)	Impact Resistance (J/m)
CE1	1%	0.33	148	126	3.3	12.2	40.26	0.387	31.73
CE2	2%	0.33	148	125	3.3	12.3	40.59	0.408	33.19
CE3	3%	0.33	148	123.5	3.2	12	38.4	0.440	36.70
CE4	4%	0.33	148	123.5	3.3	12.5	41.25	0.440	35.23
CE5	5%	0.33	148	124.5	3.3	12.3	40.59	0.419	34.06

Beyond the optimal CE3, impact resistance decreased slightly to 35.23 J/m for CE4 and 34.06 J/m for CE5. The reduction at higher filler content can be attributed to particle agglomeration and reduced matrix continuity, which create stress concentration points and facilitate crack growth [28,29]. The non-linear variation of impact resistance with filler content reflects the influence of microstructural factors, including filler dispersion and matrix-filler interfacial adhesion. At the optimal filler loading, a balance between stiffness, toughness, and energy absorption is achieved, providing the highest resistance to impact without significant compromise of the matrix integrity [30].

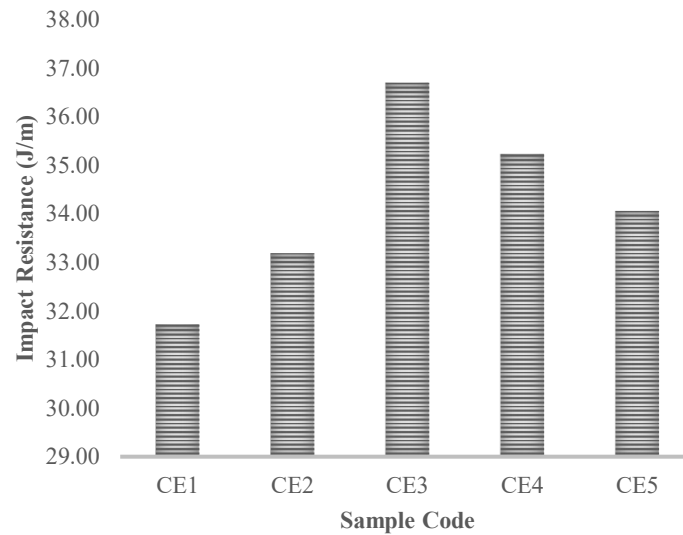


Figure 7. Impact resistance

The SEM image in Figure 8 reveals that the CaCO_3 particles possess irregular and angular shapes with rough surface textures, typical of biogenic materials that have undergone mechanical grinding and thermal annealing. This morphology contributes to improved mechanical interlocking within the polymer matrix, enhancing stiffness and toughness at lower to moderate filler loadings. However, the image also shows minor agglomerations, particularly between finer particles, which may reduce the homogeneity of filler dispersion at higher concentrations [30]. These agglomerates can create local stress concentration zones, thereby interfering with the effective transfer of stress across the matrix-filler interface. This supports the finding that while moderate filler incorporation, up to a 3% volume fraction, is beneficial for reinforcing the composite, excessive loading introduces microstructural inefficiencies that degrade mechanical performance. The morphological insight from Figure 8 thus reinforces and contextualizes the performance trends discussed in the tensile and impact analyses.

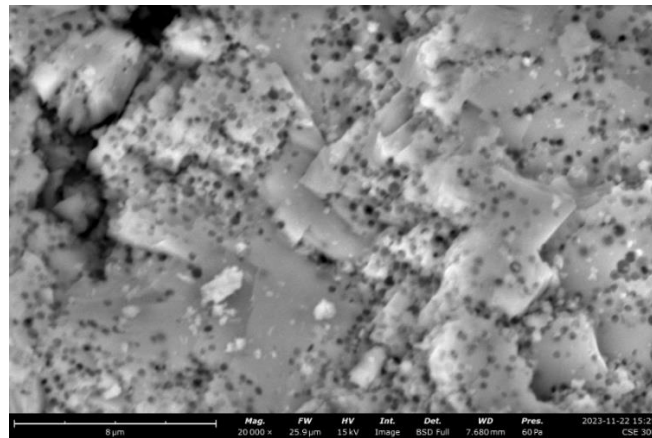


Figure 8. SEM image of CaCO_3 eggshell powder at 8 μm magnification

4 Conclusions

In conclusion, this study focused on the relatively unexplored range of low filler fractions (1-5%) in unsaturated polyester composites reinforced with calcium carbonate derived from waste eggshells. The addition of eggshell-derived filler had a significant influence on mechanical performance. The ultimate tensile strength decreased gradually with increasing filler content, reaching 19.46 MPa at 5% filler, which corresponds to a reduction of 6.7% compared with the lowest-filler composition. Conversely, the elastic modulus increased to 1.58 GPa, representing a 9.7% enhancement. The impact resistance improved with filler addition, achieving the maximum value of 91.75 J/m at 3% filler, an increase of 15.7%, followed by a slight decrease at higher fractions.

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SEM result revealed irregular particle morphology, surface roughness, and local agglomeration, which explained the balance between stiffness enhancement and strength reduction. Overall, the results indicate that 3% filler volume offers the most balanced performance, combining improved stiffness and toughness. For future work, further studies should investigate surface modification of the filler to enhance interfacial adhesion, optimize particle size distribution, and conduct long-term durability testing under various environmental conditions. Comparative investigations using other polymer matrices or hybrid biofiller systems are also recommended to broaden the application potential of this sustainable composite material.

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