Investigation of Novel Poly(urethane-urea) and MMT Foams Derived via In-situ Technique

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Abstract
Thermal and mechanical behavior and fire performance of novel series of flame-retardant high impact poly(urethane-urea) (PUU)/montmorillonite nanocomposites and foams were designed and studied by various means. Silicate layers of hydroxyl modified montmorillonite (H-MMT) was well exfoliated in PUU matrix due to in-situ reaction between the clay and poly(urethane-urea). The combination of PUU and montmorillonite modified with bis-2-hydroxyethyl ammonium as flame retardant enhanced the charring capacity and non-flammability of foams and also increased the thermal performance with nanofiller loading. FESEM illustrated increased cell density and reduced cell size in PUU/H-MMT 1-5 Foam (1-5 wt. % nanofiller) relative to pure PUU foam. Compression strength and modulus of PUU/H-MMT 1 Foam (1 wt. % nanofiller) was 30.1 MPa and 3 GPa respectively, which was increased to 36.7 MPa and 8 GPa in PUU/H-MMT 5 Foam (5 wt. % nanofiller). 10 % thermal decomposition temperature of PUU/H-MMT 1-5 Foams were in the range 488–519 °C. In PUU/H-MMT 5 Foam Tg was increased to 157 °C relative to PUU/H-MMT 1 (Tg, 147 °C). LOI and UL 94 tests had shown improved non-flammability (V-0 rating) with H-MMT loading.

Keywords: Poly(urethane-urea); montmorillonite; foam; LOI; UL 94

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1. Introduction

Polyurethanes have been categorized as high performance polymers owing to their unique physical and mechanical performance. The study of structure-property relationship in polyurethanes has gained considerable significance because of their huge relevance in various industries [1]. Nevertheless, the inherently poor thermal stability and inadequate flame retardancy of thermoplastic polyurethanes have been the major cause to limit their end use in certain areas, especially in space applications [2-4]. One of the accredited approaches for the improvement of the characteristics of polyurethane has been the chemical modification of structure by blending or copolymerization with heat constant materials. In this regard, polyurethane-polyurea has been characterized as modified two-component polyurethane systems [5, 6]. Several attempts have been made to integrate the urea units into polyurethane spine. A simple and successful way has been known as the reaction of isocyanate terminated polyurethane with amines to generate modified urea backbone [7, 8]. Polyurea systems have sometimes been characterized as modified two-component polyurethane systems capable of utilization in elastomeric coating, lining systems, protective coating and in other newest technologies [9]. The most important commercial polyurethane products are foams that are commonly classified as either flexible or rigid depending on their mechanical performance and cross-link densities. Owing to superior heat and flame resistance and mechanical properties in addition to low density, rigid polyurethane foams have been widely employed in building insulation, domestic appliances, aerospace-grade rigid foam core materials for aircraft flight decks and interior cabins, customized flame-retardant polyurethane products, etc. [10]. Owing to their important relevance, first class physical properties are demanded for these structural materials [11]. To obtain polyurethane foams with good mechanical properties, different types of hybrid or modified materials have been developed [12-14]. Adding additives to foam matrix has been the understated way to improve the characteristics of these materials. Of particular interest is the developed nanocomposite technology consisting of polymer and organically modified montmorillonite (MMT) because they often exhibit remarkably improved mechanical and various other properties compared to those of the virgin polymer. Incidentally, polymer/clay nanocomposites have been promising materials since last few decades. In order to obtain good chemical affinity between polymer and clay, the use of modified clay with high affinity has been recommended [15, 16]. Researchers have been attempting to use different type of clay functionalization [17]. Several modifications have been proposed to describe the observed improvement in flammability properties of polymers by the formation of polymer/clay nanocomposites. One of them is the reduction in heat release rate due to the formation of a protective surface barrier/insulation layer consisting of accumulated clay platelets with a small amount of carbonaceous char [18-20]. The presence of functional groups is generally known to improve the intra-gallery polymerization, which in sequence initiates improved clay dispersion [21, 22]. Currently, we have prepared poly(urethane-urea) via polycondensation of 4,4'-diphenylmethane diisocyanate, polycaprolactone triol and p-phenylene diamine. Later, in-situ polymerization of hydroxyl modified montmorillonite (H-MMT) and poly(urethane-urea), series of nanocomposites and foams have been developed. Mechanical, morphological, thermal and flame retardant properties of nanocomposites and foams were determined via field emission scanning electron microscopy (FE-SEM), thermo gravimetric analysis (TGA), dynamic mechanical thermal analysis (DMTA), tensile, limiting oxygen index (LOI) and UL 94 tests. Main objective of this paper was to find out the effect of development of in-situ polymer-clay linked systems on the physical characteristics of new
poly(urethane-urea)/clay nanocomposites and foams.

2. Experimental

2.1 Instrumentation

Fourier transform infrared (FTIR) spectra were recorded using Excalibur Series FTIR Spectrometer, Model No. FTSW 300 MX manufactured by BIO-RAD. Inherent viscosity (\(\eta_{inh}\)) was measured in THF (30 °C) with Ubbelohde viscometer using 0.5 g/dL polymer solution. The weight-average molecular weight (\(M_w\)) and polydispersity index (PDI) were calculated through gel-permeation chromatography (GPC) using THF as eluent and refractive index (RI) detector. The compression strength and modulus were measured with a universal electronic tensile machine (Shimadzu, Japan) at a compressive rate of 2 mm/min according to ASTM D 1621-94. Mechanical properties were estimated using standard procedures and formulae. For phase morphological studies, samples were cryogenically fractured in liquid nitrogen and the morphology was investigated by FEI Nova 230 field emission scanning electron microscope (FE-SEM). For transmission electron microscopy (TEM) measurement, samples were prepared at -50 °C with an Ultracut E ultramicrotome by Reichert and Jung using a diamond knife. Measurements were carried out on a LEO 912 Omega (120 kV). Thermal stability of the nanocomposites was determined by NETZSCH thermogravimetric analyzer (TGA), model no. TG 209 F3, using 1-5 mg of the sample in Al₂O₃ crucible from upto 800 °C at a heating rate of 10 °C/min under nitrogen flow rate of 30 mL/min. The dynamic mechanical thermal analysis was performed in the temperature range 0-300 °C with DMTA Q800 (frequency of 5 Hz, heated at 10 °C/min). LOI value was measured on samples (100×5.8×3 mm³) according to the standard oxygen index test ASTM D2863-77 via FTA IL. UL-94 test was performed (110×10.1 ×3 mm³) according to ASTM D635-77 for UL-94 test. Moisture absorption test was carried out according to ASTM C272 standards. Test specimens were prepared three inch by three inch by 0.5 inch thickness. After weighing, the test samples were placed into deionized water for given period of time (24, 48, 72 & 96 h). The specimens were removed from water and weighed for the assessment of water absorption percentage. The increase in mass was calculated as:

Increase in weight (%) = (W – D)/D \times 100

Where W = Wet weight; D = Dry weight

2.2 Materials

Polycaprolactone triol (average \(M_n\) ~900), \(p\)-phenylene diamine (97%), tetrahydrofuran (THF, 99 %) and 1,3,5-triacyloylhexahydro-1,3,5-triazine (98%, surfactant for foaming) were provided by Aldrich. 4,4’-Diphenylmethane diisocyanate (39.4%) i.e. Lupranate M-20S, polymethylene polyphenyl isocyanate, was supplied by BASF. A mixture of cyclopentane and isopentane (70:30) was used as the blowing agent. MMT-OH (Cloisitew 30B, montmorillonite modified by bis-2-hydroxyethyl ammonium) supplied by Southern Clay Products.
2.3 Synthesis of poly(urethane-urea)

4,4’-Diphenylmethane diisocyanate was dehydrated at 80 °C for 24 h. Polycaprolactone triol was dehydrated at 60 °C for 24 h. Afterwards, the polyol and diisocyanate were sonicated in 10 mL THF (NCO/OH=1) with steady stirring of 4h. p-Phenylenediamine (equimolar amount) was dissolved separately in 10 mL of THF with constant stirring of 4h. The above mixtures were then mixed and refluxed at 80 °C for 12 h. Later, the mixture was cast in Teflon mold and cured at 80 °C to obtain PUU film (Scheme 1). \( \eta_{inh} = 1.67 \text{ dLg}^{-1} \); \( M_w = 42 \times 10^2 \text{ gmo}l^{-1} \); PDI = 2; % Yield = 89 %.

![Scheme 1 Schematic preparation of poly(urethane-urea).](image)

2.4 Synthesis of PUU/MMT-OH nanocomposites and foams

Akin to the preparation of poly(urethane-urea), dehydrated 4,4’-diphenylmethane diisocyanate, polycaprolactone triol and p-phenylene diamine were mixed in THF. Desired amount of dehydrated clay content (1-5 %) was added depending upon the weight of PUU prepared. Afterward, the above prepared mixture was refluxed at 80 °C for 8 h. For reactive foaming, 1,3,5-triacyloylhexahydro-1,3,5-triazine was
used as surfactant (1 wt.% and pentanes were employed as blowing agent (5 wt.%). The above foaming mixture with all the ingredients was mixed using impeller at 4000 rpm and foaming occurred in a closed plastic container. The foams were then cured at 80 °C for 8 h.

3. Results and Discussion

3.1 FTIR Analysis

FT-IR spectrum of PUU is specified in Fig. 1A. Carbonyl peaks for anhydride and urethane were emerged at 1721, 1742 and 1782 cm\(^{-1}\). NHCOO stretching and bending vibrations also appeared at 3222 and 1596 cm\(^{-1}\) respectively. The aliphatic and aromatic protons were observed near 2922 and 3055 cm\(^{-1}\). C–O stretching vibration was found at 1300 cm\(^{-1}\). PUU/H-MMT 1 Foam (Fig. 1B) displayed nearly all characteristic bands of polyurethane found in the consequent spectrum. Manifestation of hydroxyl stretching vibration at 3412 cm\(^{-1}\) was allied with the unreacted functionalities of modified MMT. N–H stretching and bending vibrations were transpired at 3220 and 1595 cm\(^{-1}\) respectively. Equivalent to PUU spectrum, the aliphatic and aromatic protons were experiential near 2912 and 3021 cm\(^{-1}\). The carbonyl functionalities appeared at 1718, 1750 and 1786 cm\(^{-1}\). Moreover, PUU/H-MMT 1 Foam displayed peaks related to C–O at 1299 cm\(^{-1}\). FTIR spectrum of PUU/H-MMT 1 Foam also revealed sharp peak at 720 cm\(^{-1}\) due to SiO\(_4\) vibration. The strong doublets owing to Si(Al)–O near 536 cm\(^{-1}\) and Si(Al)–O–Si peak at 1110 cm\(^{-1}\) were observed in the spectrum because of the integration of montmorillonite in the matrix [23].

![Figure 1 FTIR spectra of (A) PUU; (B) PUU/H-MMT 1.](image-url)
3.2 Compressive strength and modulus of PUU and nanocomposite Foams

The compressive strength and modulus of neat poly(urethane-urea) are presented in Fig. 2A and B. Table 1 has shown that the compressive strength and modulus of foams increased progressively with the increase in functionalized clay content from 1 to 5 wt. %. [24]. Results have demonstrated that neat PUU Foam had compressive strength and modulus of 22.5 MPa and 0.6 GPa respectively. In PUU/H-MMT 1 Foam, addition of 1 wt. % H-MMT content in the matrix improved the compressive strength and modulus values 30.1 MPa and 3 GPa respectively. Further inclusion of 3 wt. % functional nanoclay raised the compressive strength and modulus values to 32.5 MPa and 6 GPa for PUU/H-MMT 3 Foam. Addition of modified layered silicate content upto 5 wt % yielded the compressive strength and modulus to reach the maximum values of 36.7 MPa and 8 GPa respectively. The continuous increase in the compressive strength of foams with clay loading clearly indicated the uniform dispersion of the clay platelets in poly(urethane-urea) matrix. It seemed that during the compression of nanocomposites foams, the external stress was transferred from the resin to layered silicate particles which were chemically linked with the PUU. General research has also shown that the fracture of materials occurs on the interface between the additives and resin. Increase in the clay content (upto 5 wt. %) has obviously increased the interface area and more energy was needed to destroy PUU/layered silicate assembly [25]. Moreover, there exist numerous gaps between platelets of layered silicate sheets. Since the compatibility of layered silicate and PUU/H-MMT was increased by the in-situ polymerization, that increased the concord and integrity of the cells causing consistent cellular foam structure.

Figure 2A Compression strength of PUU Foam and PUU/H-MMT 1-5 Foams.
Figure 2B  Compression Modulus of PUU Foam and PUU/H-MMT 1-5 Foams.

Table 1  Mechanical properties of PUU and PUU/H-MMT nanocomposites and foams.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Compression strength (MPa)</th>
<th>Compression Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PUU Foam</td>
<td>22.5</td>
<td>0.6</td>
</tr>
<tr>
<td>PUU/H-MMT 1 Foam</td>
<td>30.1</td>
<td>3</td>
</tr>
<tr>
<td>PUU/H-MMT 3 Foam</td>
<td>32.5</td>
<td>6</td>
</tr>
<tr>
<td>PUU/H-MMT 5 Foam</td>
<td>36.7</td>
<td>8</td>
</tr>
</tbody>
</table>
3.3 Morphology investigation

Scanning electron microscopy was used to study poly(urethane-urea) foam and PUU-based nanocomposites and foam samples as shown in Fig. 3A-F. Fig. 3A and B show the morphology of PUU/H-MMT 1 and PUU/H-MMT 3 nanocomposites. Micrograph in Fig. 3A illustrated layered but homogeneous morphology with consistent dispersion of clay particles in poly(urethane-urea) structure. Fig. 3B also depicted somewhat uniform nanoclay dispersion, but homogeneity was not as marked for Fig. 3B. Morphology of neat PUU foam was entirely different than PUU/H-MMT 1 and PUU/H-MMT 3 nanocomposites. Fig. 3C showed the configuration of particularly large cells in Fig. 3C. PUU/H-MMT 1-5 Foams (Fig. 3 D-F) also formed some sort of cellular structure in which cell size was not so larger when compared with neat foams. There was instantaneous transformation in cell density and size upon in-situ polymerization with H-MMT (nearly 100-fold decrease) in PUU/H-MMT 1-5 Foams. Distorted square and oval shapes were visible in the micrograph (Fig. 3F). The observation of the micrographs has depicted the difference between PUU/H-MMT 1-5 Foam morphologies containing clay nanoparticles and PUU foam. The results have suggested that there was a tremendous effect of surface chemistry on foam morphology. Furthermore, in PUU/H-MMT 1-5 Foams, several granular particles were found inside the cells. The average cell size was found to increase in PUU/H-MMT 5 Foam, however, the cell size and density of PUU/H-MMT 1 Foam was analogous to PUU/H-MMT 3. Over all results suggested that the dense cellular morphology with dispersed clay particles was liable for the enhanced mechanical behavior of novel PUU/H-MMT 1-5 Foams.

3.4 Thermogravimetric analysis

Thermal data obtained through TGA of PUU and PUU/H-MMT nanocomposites and foams are presented in Table 2. Initial weight loss (T0) was observed at 421 ºC, 10 % degradation temperature (T10) at 434 ºC and maximum decomposition temperature (Tmax) was found at 456 ºC for neat poly(urethane-urea). The char yield of PUU was 29 %. In the case of nanocomposites, PUU/H-MMT 1 nanocomposite (1 wt. % of clay) showed T0 = 447 ºC; T10 = 459 ºC; Tmax = 494 ºC and char yield = 32 %. PUU/H-MMT 3 (T0 = 464 ºC; T10 = 478 ºC; Tmax = 500 ºC and char yield = 36 %) and PUU/H-MMT 5 (T0 = 485 ºC; T10 = 498 ºC; Tmax = 521 ºC and char yield = 39 %) nanocomposites showed continuing increase in thermal stability with layered silicate loading of 3 and 5 wt. %. TGA results of PUU/H-MMT 1-5 Foams showed appreciable difference in wt. % loss temperature relative to neat PUU and PUU/H-MMT 1-5 nanocomposites. However, in both the series (nanocomposites and foams), the higher thermal stability was observed for 5 wt. % clay loaded sample i.e. PUU/H-MMT 5 nanocomposite and PUU/H-MMT 5 foam (Fig. 4). Among the foams, PUU/H-MMT 1 Foam obtained from PUU and 1 wt. % H-MMT, materials was found to have T0 of 488 ºC, T10 of 495 ºC and Tmax of 556 ºC. Adding up 3 wt. % H-MMT in PUU/H-MMT 3 Foam further enhanced the heat constancy to T0, T10 and Tmax of 496 ºC, 512 ºC and 579 ºC respectively. PUU/H-MMT 5 Foam had still higher values of T0, T10 and Tmax around 519 ºC, 539 ºC and 590 ºC respectively. Moreover, the weight of residue at 600 ºC was in the range of 43-62 % for PUU/H-MMT 1-5 Foam, indicating higher thermal stability with rising H-MMT content in foams. The in-situ reaction between the nanofiller and matrix actually improved the dispersion of clay in PUU and intern enhanced the thermal stability of the nanocomposites and foams. Loss tangent (tan δ) vs. temperature plots from DMTA was used to study glass transition, as shown in Fig. 5. Addition of H-MMT increased the segmental Tg of PUU from 118 ºC to 139
ºC in PUU/H-MMT 5 nanocomposite and to 157 ºC in PUU/H-MMT 5 Foam. Glass transition of PUU/H-MMT 1-5 nanocomposites and PUU/H-MMT 1-5 Foams was also increased with clay loading with in the series. Over all thermal properties of PUU/H-MMT 1-5 Foams were found to be superior than reported materials [26].

Figure 3 FESEM images of (A) PUU/H-MMT 1; (B) PUU/H-MMT 3; (C) Neat PUU Foam; (D) PUU/H-MMT 1 Foam; (E) PUU/H-MMT 3 Foam; (F) PUU/H-MMT 5 Foam.
Table 2  Thermal analyses data of PUU and PUU/H-MMT nanocomposites and foams.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$T_g$ (°C)</th>
<th>$T_0$ (°C)</th>
<th>$T_{10}$ (°C)</th>
<th>$T_{\text{max}}$ (°C)</th>
<th>$Y_c$ at 600 °C (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PUU</td>
<td>118</td>
<td>421</td>
<td>434</td>
<td>456</td>
<td>29</td>
</tr>
<tr>
<td>PUU/H-MMT 1</td>
<td>130</td>
<td>447</td>
<td>459</td>
<td>494</td>
<td>32</td>
</tr>
<tr>
<td>PUU/H-MMT 3</td>
<td>135</td>
<td>464</td>
<td>478</td>
<td>500</td>
<td>36</td>
</tr>
<tr>
<td>PUU/H-MMT 5</td>
<td>139</td>
<td>485</td>
<td>498</td>
<td>521</td>
<td>39</td>
</tr>
<tr>
<td>PUU/H-MMT 1 Foam</td>
<td>147</td>
<td>488</td>
<td>495</td>
<td>566</td>
<td>43</td>
</tr>
<tr>
<td>PUU/H-MMT 3 Foam</td>
<td>153</td>
<td>496</td>
<td>512</td>
<td>579</td>
<td>45</td>
</tr>
<tr>
<td>PUU/H-MMT 5 Foam</td>
<td>157</td>
<td>519</td>
<td>539</td>
<td>590</td>
<td>62</td>
</tr>
</tbody>
</table>

$T_g$: Glass transition temperature  
$T_0$: Initial decomposition temperature  
$T_{10}$: Temperature for 10% weight loss  
$T_{\text{max}}$: Maximum decomposition temperature  
$Y_c$: Char yield; weight of polymer remaine

Figure 4  TGA curves of PUU/H-MMT Foams at 10 °C/min (N₂).
Table 3 shows the effect of hydroxy-clay loading on the limiting oxygen index (LOI) value and UL-94 rating of PUU/H-MMT 1-5 Foams. Results have established that adding up 1 wt. % clay in PUU/H-MMT 1 Foam showed higher LOI value of 41 % relative to pure PUU (31 %). Further addition of 3 wt. % nano-clay in PUU/H-MMT 3 Foam improved the LOI value to 44 %. PUU/H-MMT 5 Foam (5 wt. % nano-modified clay) led to LOI value of 47 %. As a result, the inclusion of 1-5 wt. % nano-clay in PUU foams augmented LOI values from 41 to 47 %, which was a noteworthy increase. LOI data thus exposed that the materials were adequately flame retardant. PUU/H-MMT 1-5 Foams were also experienced for UL-94 tests. PUU/H-MMT Foams operated fine in UL-94 tests indicating that the amount of nanofiller was sufficient to bring superior flame retardancy. The reason for the superior flame retardancy of materials was the synergistic effect of clay and PUU in foams due to chemical bonding between the components facilitating V-0 rating. In vertical UL-94 testing, a stiff char layer was formed over the foam surface shielding the material underneath [27]. Clay introduction in PUU has generated a very smooth, compact, continuous and heat-resistant charred residue layer upon thermal degradation in air and in flame. The charred residue layer cover over the foam surface formed an insulating barrier between the gaseous flame area and the bulk of the materials.
Table 3 LOI values and UL-94 test results of PUU/H-MMT Foams.

<table>
<thead>
<tr>
<th>Sample</th>
<th>LOI (%)</th>
<th>UL-94</th>
</tr>
</thead>
<tbody>
<tr>
<td>PUU</td>
<td>31</td>
<td>V-0</td>
</tr>
<tr>
<td>PUU/H-MMT 1 Foam</td>
<td>41</td>
<td>V-0</td>
</tr>
<tr>
<td>PUU/H-MMT 3 Foam</td>
<td>44</td>
<td>V-0</td>
</tr>
<tr>
<td>PUU/H-MMT 5 Foam</td>
<td>47</td>
<td>V-0</td>
</tr>
</tbody>
</table>

3.6 Moisture absorption test for PUU/H-MMT Foams

Weighed foam samples were immersed in de-ionized water for 24-96 h at 30 °C for moisture absorption test. After the desired time period, the specimens were taken out from water, dried and weighed. Table 4 illustrates the % water absorption as percentage of weight gained by the foams. It was found that PUU/H-MMT 5 Foam showed maximum percentage of moisture absorption of 3.65 % after 96 h over the period of time. For PUU/H-MMT 1 Foam had lower percentage of moisture absorption around 3.14 % (96 h) and PUU/H-MMT 3 Foam depicted value at 3.25 % (96 h). Nevertheless, the water absorption capacity was gradually amplified with the time for all the PUU/H-MMT 1-5 Foams prepared.

Table 4 Water absorption of PUU/H-MMT 1-5 Foams.

<table>
<thead>
<tr>
<th>Time (h)</th>
<th>PUU/H-MMT 1 Foam</th>
<th>PUU/H-MMT 3 Foam</th>
<th>PUU/H-MMT 5 Foam</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>0.62</td>
<td>1.11</td>
<td>1.39</td>
</tr>
<tr>
<td>48</td>
<td>1.88</td>
<td>2.05</td>
<td>2.38</td>
</tr>
<tr>
<td>72</td>
<td>2.15</td>
<td>2.55</td>
<td>3.22</td>
</tr>
<tr>
<td>96</td>
<td>3.14</td>
<td>3.25</td>
<td>3.65</td>
</tr>
</tbody>
</table>
4. Conclusions

In this work, our foremost aim was to set up novel modified PUU/clay-based nanocomposites and foams. The influence of H-MMT modification and loading on the compression strength and modulus, morphology, thermal and flammability properties was investigated in feature. The nano-dispersion of H-MMT via in-situ reaction resulted in nonflammable poly(urethane-urea)/clay nanocomposite with enhanced thermal and mechanical properties. The modified foam materials demonstrated significantly reduced cell size structure, relative to neat PUU foam owing to well-dispersed montmorillonite. Main success was the attainment of LOI values between 41-47 % and V-0 rating of PUU/H-MMT 1-5 Foams. A stable residue layer was formed over the foam surface during vertical UL-94 test. The layer actually cut off the heat transfer and mass exchange between the gaseous flame area and underlying polymer composite foam and so improved the fire resistance of material. New foam materials also had fine water absorption capability. Novel poly(urethane-urea)/organoclay foams may act as energy saving materials for building heating/cooling systems with improved heat-insulating ability, heat insulation systems and aerospace parts.

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