

## Emulsion Polymers of Carboxymethyl Cellulose and Methyl Methacrylate with Sodium Dodecyl Sulfate Surfactants as Soil Stabilizer

Akhmad Zainal Abidin\*, R. B. Emil Trisatya Nuriman, Narendra Afian Pradipto, Ridwan P. Putra

Department of Chemical Engineering, Faculty of Industrial Technology, Institut Teknologi Bandung,  
Jalan Ganesha 10, Bandung, 40132, Indonesia

\*Corresponding Author: [zainal@che.itb.ac.id](mailto:zainal@che.itb.ac.id)

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### Abstract

Polymer-based materials have gained considerable attention in the road construction sector due to their ability to improve the mechanical strength and durability of soil. In this study, carboxymethyl cellulose-methyl methacrylate (CMC-MMA) emulsion polymers were prepared from different MMA to CMC mass ratios with sodium dodecyl sulfate surfactant. The synthesized materials were homogeneously mixed with laterite soil, and their unconfined compressive strengths were measured using dry and wet methods on a universal testing machine. The results indicated that the polymers were obtained in high yields with optimum solids content. Characterization using Fourier transform infrared confirmed the homopolymerization probability of monomers in the materials with increased MMA fractions. In addition, the viscosity of the graft-copolymers measured at room temperature was in the range of 1.2-3.2 cP. The polymer-stabilized soils exhibited maximum unconfined compressive strengths of 564 and 162 psi, respectively, obtained from the dry and wet methods. In general, the rise of MMA fraction in the treated soils enhanced the compressive strength of the soil, with a slight increase in soil pH. This study unfolds the potential of cellulose-based materials that can be implemented in soil reinforcement technology.

*Keywords:* Carboxymethyl cellulose, copolymerization, methyl methacrylate, soil stabilization, unconfined compressive strength

### INTRODUCTION

Urban roads serve as a major part of the city transportation system that their construction and improvement are vital for the advancement of urban development and human well-being (Wen, Gui, Zhang, & Hui, 2020). Many road-building materials and energy are required during the construction of roads and highways (Diez et al., 2016). Adequate planning and design strategies are necessary for the long term sustainability (Kuenzel, Mueller, Teizer, & Blickle, 2015). In addition, some maintenance and rehabilitation efforts are also needed to ensure the durability and functionality of the infrastructures (X. Zhang & Gao, 2012).

Soil stabilization employing a stabilizing material offers a scalable and straightforward approach to constructing roads without asphalt coverings. This method is considered low-cost with fewer unfavorable effects compared to the paving technique (Jin, Tan, Liu, Zheng, & Zhang, 2019). The soil is stabilized to enhance its mechanical strength and durability, and prevent erosion and dust formation (Naeini, Naderinia,

& Izadi, 2012). The improved soil properties are due to the interaction of the stabilizing material with soil voids. The material fills the interparticle spaces and forms interconnection bridges within the soil, making this technique promising in soil reinforcement technology (Liu et al., 2019).

In recent years, polymer materials have been studied as soil stabilizing agents to improve the unconfined compressive strength and bearing capacity of soils (Huang, Kogbara, Hariharan, Masad, & Little, 2021). Kolay, Dhakal, Kumar, & Puri, (2016) investigated the effect of liquid acrylic polymer to stabilize fine-grained soils. The study shows that the unconfined compressive strength of soil prepared at optimum moisture content increases from 30 to 75% in an open-air environment. Soil reinforcement using sodium carboxymethyl cellulose also indicates enhanced compressive strength, tensile strength, and Young's modulus of the amended soils (Ma & Ma, 2019). The water-retaining property, anti-erosion resistance, strength, and durability of soils are also improved with the increase of vinyl acetate polymer fraction in the soil (Song et al., 2019). The results

encouraged us to synthesize other functional polymers that are potential to be implemented in the road construction sector.

In this work, carboxymethyl cellulose (CMC) and methyl methacrylate (MMA) were used to prepare CMC-MMA emulsion polymers. Cellulose-based material was opted as a scaffold backbone of the polymers due to its wide range of applications (Umaningrum et al., 2021, Souhoka & Latupeirissa, 2018, Ningsih, Ariyani, & Sunardi, 2019). Sodium dodecyl sulfate (SDS) was employed as a surfactant. The graft-copolymerization process was carried out with different MMA to CMC mass ratios. Fourier transforms infrared (FTIR) spectroscopy was employed to confirm the CMC-MMA synthesis. The synthesized CMC-MMA emulsion polymers were used to stabilize laterite soil, and the polymer-stabilized soils were measured for their unconfined compressive strengths. This study is the first to report the grafting of CMC-MMA copolymers with SDS surfactant as a potential soil stabilizing agent.

## METHODOLOGY

### Materials and Instruments

Carboxymethyl cellulose (CMC,  $C_8H_{16}NaO_8$ ), sodium dodecyl sulfate (SDS,  $CH_3(CH_2)_{11}SO_4Na$ ), methyl methacrylate (MMA,  $C_5H_8O_2$ ), and potassium persulfate (KPS,  $K_2O_8S_2$ ) were used as received without further purification. Laterite soil was obtained from Sukabumi, West Java, Indonesia. Distilled water was used throughout the experiments. FTIR spectra were acquired on an IRPrestige-21 Shimadzu. An Ostwald viscometer was used to measure the viscosity of emulsion polymers. The unconfined compressive strength of soil was tested on a universal testing machine.

### Synthesis of Emulsion Polymers

MMA and CMC with a mass ratio of 4.44, 6.67, 8.89, 11.11, and 13.33, respectively, were mixed in a four-neck flask equipped with a stirrer, a condenser, and a thermometer. Afterwards, distilled water was added by adjusting the maximum monomer concentration to 40% (w/w). SDS was subsequently poured into the mixture by controlling the CMC to SDS mass ratio to 10:1. Here, SDS acted as the surfactant. The mixture was heated to reach a temperature of 70 °C. KPS was introduced to the mixture dropwise as a chemical initiator until a KPS to SDS mass ratio of 0.56 was attained. The reaction was then allowed to proceed for 4 h until an emulsion was formed.

### Solids Content Analysis

As much as 1 g of the synthesized emulsion polymer was poured into an evaporating dish. The dish was heated up at 105 °C until a constant weight was reached, indicating the removal of the solvent in the polymer. The dried polymer was weighed, and the experimental solids content,  $SC_{exp}$ , was calculated using Equation (1).

$$SC_{exp}(\%) = \frac{\text{weight}_{\text{dried polymer}}}{\text{weight}_{\text{emulsion}}} \times 100\% \quad (1)$$

The yield obtained in this study was determined from the ratio of  $SC_{exp}$  to theoretical solids content,  $SC_{theo}$ , as justified in Equation (2).

$$\text{Yield}(\%) = \frac{SC_{exp}}{SC_{theo}} \times 100\% \quad (2)$$

### Characterization of Emulsion Polymers

The viscosity of the emulsion polymers was measured at room temperature with distilled water as the reference. The infrared spectra were recorded over the range of 500-4500  $cm^{-1}$ .

### Unconfined Compressive Strength Tests

The compression test was implemented according to ASTM standard C873 for concrete materials. The unconfined compressive strength was defined as the maximum load that a sample could withstand before it collapsed. Prior to the measurements, the as-synthesized emulsion polymers were homogeneously mixed with the soil to give a 4% (w/w) of polymer content in the mixture (ASTM standard D698-07). The mixtures were shaped into cylindrical specimens. The molded soil samples were allowed to dry at room temperature for seven days. The unconfined compressive strength ( $\sigma$ ) was obtained from the ratio of the maximum load ( $P$ ) to the cross-sectional area of sample ( $A$ ), as stated in Equation (3).

$$\sigma = \frac{P}{A} \quad (3)$$

Two methods were used to measure the unconfined compressive strength of the polymer-stabilized soils: the dry method and the wet method. The prepared specimens were directly measured for their unconfined compressive strength in the dry method. The wet method required the immersion of the specimens in water for 3 minutes followed by curing for 5 minutes before measuring the unconfined compressive strength of the soil (Zandieh & Yasrobi, 2010).

### Soil pH Measurements

The soil pH stabilized with the emulsion polymers was measured using a digital pH meter according to ASTM standard D4972. The pH meter was calibrated using a reference solution prior to the measurements.

## RESULTS AND DISCUSSION

### Solids Content in Emulsion Polymers

The solids content in all emulsion polymers synthesized with different MMA to CMC mass ratios is shown in Figure 1. The results indicated that the rise of MMA fraction in the mixture tended to increase the solids content in the emulsion polymer. The increase of MMA is likely to induce the formation of chemical bonds between the monomers, resulting in the high solids content in the products (Han, Shao, Wang, Guo, & Liu, 2010). The experimental solids content was close to the theoretical values, suggesting that almost all monomers were crosslinked during the reaction.

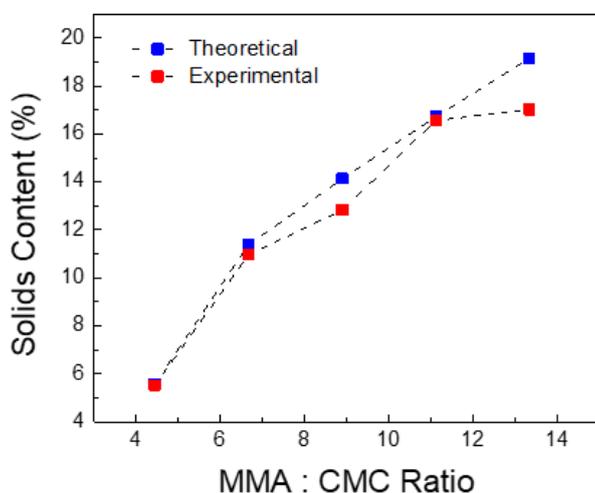


Figure 1. Solids content in the emulsion polymers prepared from different MMA: CMC mass ratios.

The experimental and theoretical solids content were used to estimate the yield of the products, as summarized in Table 1. The results showed that nearly all yields obtained from different MMA to CMC ratios are higher than 90%. A slightly lower yield of 88.85% was observed in the product with an MMA to CMC ratio of 13.33. This was expected due to the homopolymerization of MMA monomers in the solution with a high MMA to CMC mass ratio. According to a study was conducted by Sadeghi, Ghasemi, & Soliemani, (2012) on graft copolymerization of methacrylamide monomer onto CMC, the homopolymerization probability of methacrylamide increases at high methacrylamide values, resulting in the decrease of graft efficiency. The

increase in the viscosity of the mixture with high monomer concentration, which inhibits free radical movement, is also responsible for the less efficient initiation during graft copolymerization (Badwaik et al., 2016). The probability of chain transfer to monomer molecules in the excess monomer concentration also reduces graft copolymerization products (Mukherjee et al., 2017).

Table 1. Yields of emulsion polymers prepared from different MMA: CMC mass ratios.

MMA: CMC	Yield (%)
4.44	99.07
6.67	96.35
8.89	90.75
11.11	99.05
13.33	88.85

### Fourier Transform Infrared Spectra

The FTIR spectra of the emulsion polymer prepared with an MMA to CMC mass ratio of 13.33 are depicted in Figure 2. A broad peak observed around 3338-3400  $\text{cm}^{-1}$  was assigned to the -OH stretching vibration of CMC (Bao, Ma, & Li, 2011). The peak detected around 2953-2999  $\text{cm}^{-1}$  was characteristic of the vibration elongation of the methylene groups (Khiari, Mhenni, Belgacem, & Mauret, 2011). A relatively low peak noticed at 2053  $\text{cm}^{-1}$  was expected due to the interaction of negatively charged sulfonate functional group of SDS, which acted as a surfactant, with the negatively charged graft copolymers that interacted through hydrophobic interactions (Ogunlusi, Ige, & Owoyomi, 2014).

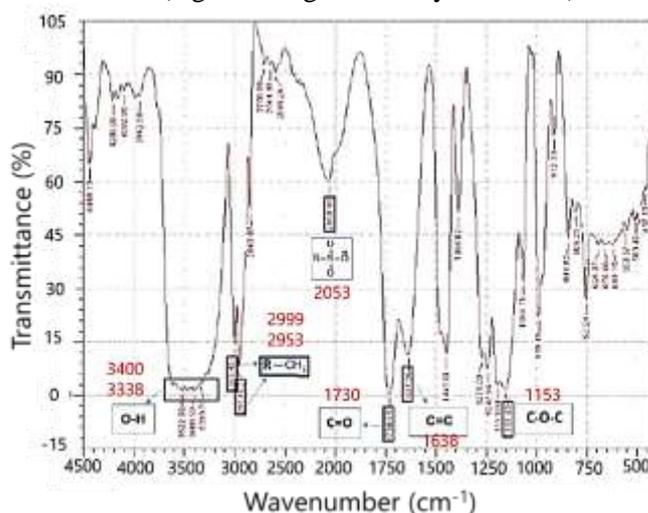


Figure 2. FTIR spectra of emulsion polymer prepared with an MMA to CMC mass ratio of 13.33

The peak observed at  $1730\text{ cm}^{-1}$  corresponded to the carbonyl (C=O) stretching in MMA (Hill et al., 2019). In comparison to the FTIR spectra of pure MMA, the carbonyl band of our graft polymer was slightly shifted. This was likely due to the polymerization reaction of C=C bonds, resulting in the reduced stretching energy of the C=O bond. The C=C bond in MMA was still identified at  $1638\text{ cm}^{-1}$ , indicating the presence of unreacted MMA monomers and homopolymerization products of MMA (Dzulkurnain, Hanifah, Ahmad, & Mohamed, 2015). The  $1153\text{ cm}^{-1}$  band was associated with the ether bonds in CMC (Fekete, Borsa, Takács, & Wojnárovits, 2016). It can be seen that a shoulder peak was present at  $1193\text{ cm}^{-1}$ . The shoulder was assigned to the ether bond formed during the graft-copolymerization of CMC-MMA monomers.

### Viscosity of Emulsion Polymers

Figure 3 plots the relationship between the viscosity of the emulsion polymers and the mass ratio of MMA to CMC used to prepare the emulsion polymers. The graph shows that the emulsion polymer prepared with a higher MMA to CMC ratio tended to produce emulsion polymers with higher viscosity. The viscosity increase was likely due to the rise of the solids content in the emulsion polymer as a result of more MMA monomers reacting to CMC monomers. (Figure 1).

In another study on starch nanoparticles emulsion polymer latexes, the increase of solids content from 40 to 50% causes the rise of emulsion viscosity from 65-88 to 169-340 mPa·s (Y. Zhang, Cunningham, Smeets, & Dube, 2019). Dastjerdi, Cranston, & Dubé, (2017) studied the synthesis of poly(n-butyl acrylate/methyl methacrylate)/CNC latexes. The results indicate that the increase of CNC loading is likely to increase the viscosity of the nanocomposite latexes. This is expected due to a thickening effect from intermolecular interactions between the hydroxyl groups on the CNC surface and carbonyl groups of the acrylic polymer. The employment of KPS as a chemical initiator also contributes to the increased ionic strength and reduce the thickness of the electrical double layer covering the surface of the polymer. A significant increase in the mixture viscosity is also noticed at the initial stages of polymerization (Yang et al., 2012).

In this study, the viscosity of the CMC-MMA emulsion polymers prepared with different MMA to CMC mass ratios was in the range of 1.23 to 3.16 cP (Figure 3). Although the viscosity of CMC is high (450 cP at  $25\text{ }^{\circ}\text{C}$  for 2% CMC solution), the measured

viscosity of our emulsion polymers is significantly lower (Salama, Etri, Mohamed, & El-Sakhawy, 2018). This was due to the fact that higher MMA fractions contributed to the reduced viscosity of emulsion polymers. From the literature, pure MMA has a viscosity of 0.554 cP at  $25\text{ }^{\circ}\text{C}$  (Fan, Zhou, Sun, & Zhang, 2009).

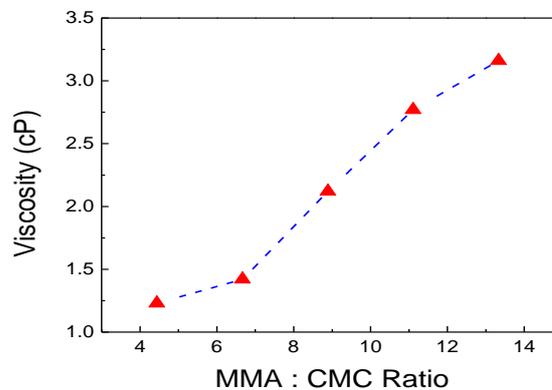


Figure 3. Viscosity of emulsion polymers at room temperature prepared with different MMA: CMC mass ratios.

### Unconfined Compressive Strength of Polymer-stabilized Soil

The soil was stabilized with CMC-MMA emulsion polymers in order to improve the unconfined compressive strength of the soil. The unconfined compressive strengths of the polymer-stabilized soils were measured using dry and wet methods. The relationship between the unconfined compressive strengths of the polymer-stabilized soils and the MMA to CMC mass ratio used to prepare the emulsion polymers measured using the dry method is depicted in Figure 4. The results were compared with the unconfined soil compressive strength without adding polymers.

From the results, the untreated laterite soil showed an unconfined compressive strength of 349 psi (2.41 MPa) measured using the dry method. The soil stabilized with CMC-MMA emulsion polymer showed enhanced unconfined compressive strength. This was expected due to the adsorption of polymer emulsion, which binds the soil particles, thus strengthening the interparticle contacts. The soil strengthening mechanism using the materials is illustrated in Figure 5. Our emulsion polymers are categorized as anionic polymers that can induce the formation of Van der Waals forces and hydrogen bonds with negatively charged soil particles (Lei, Lou, Li, Jiang, & Tu, 2020). Compared to the electrostatic interactions, these interactions are relatively weaker. In our study, the anionic polymers are stabilized with the negatively

charged SDS surfactant through hydrophobic interactions with the alkyl chains (Laguerre et al., 2015). The positively charged sodium ions in the SDS surfactant acted as a bridge between the anionic CMC-MMA polymers and negatively charged soil particles.

In another study conducted by Hataf, Ghadir, & Ranjbar, (2018) on soil stabilization using chitosan biopolymer, incorporating chitosan into clay soil improves the cohesion between the particles enhances the mechanical properties of the media. A similar finding was also reported by (Liu et al., 2017) on the effect of mixed polyurethane organic polymer and polypropylene fiber stabilization on the compressive strength of sand. The study reveals that polyurethane can improve the structural stability of sand with the best composition attained using 4% polymer and 0.2-0.3% fiber. The sand and fiber are tightly bound by the polymer membrane, which enhances the cohesion and strength of the sand.

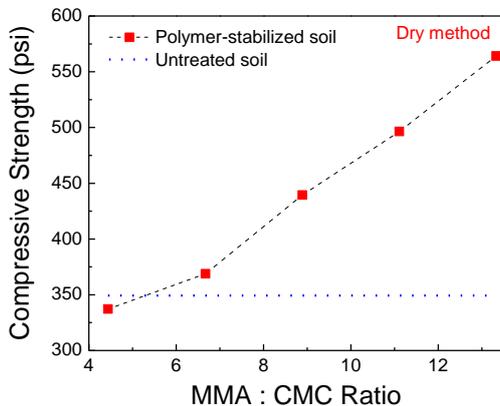


Figure 4. Unconfined compressive strengths of soils stabilized with emulsion polymer prepared with different MMA: CMC mass ratios measured using the dry method.

Our study showed that the unconfined compressive strength of polymer-stabilized soil was likely to increase with the rise of MMA fraction in the polymer. The increase of solids content in the emulsion polymer with high MMA monomers was responsible for the improved unconfined compressive strength. Only the soil stabilized with emulsion polymer prepared with MMA to CMC mass ratio of 4.44 exhibited lower unconfined compressive strength than the untreated soil.

This phenomenon was associated with the high moisture content in the emulsion polymer, which can break the bonds between adjacent chains of the polymer, resulting in the reduced mechanical strength of the soil (Kosmela et al., 2019). The soil stabilized with CMC-MMA emulsion polymers was measured

for its unconfined compressive strength using the dry and wet methods.

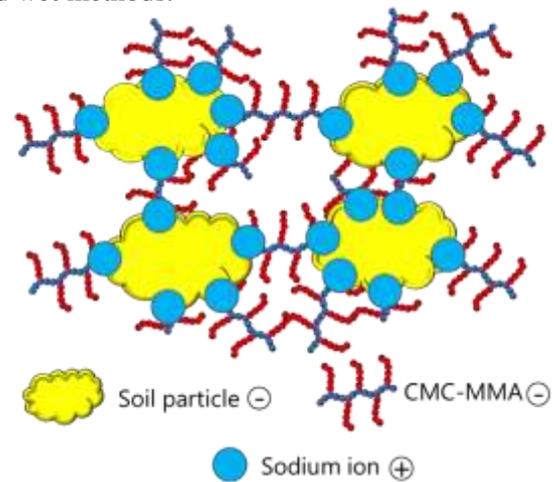


Figure 5. Scheme of soil strengthening mechanism using CMC-MMA polymer emulsions.

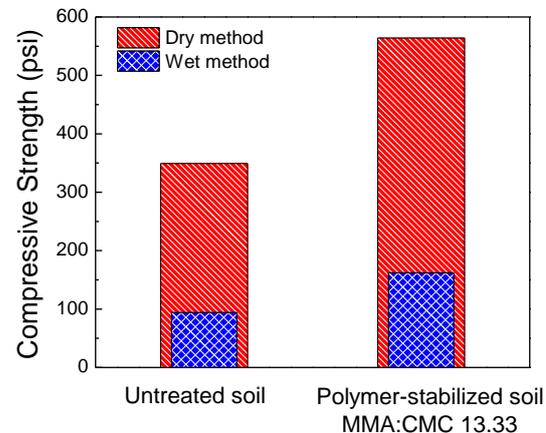


Figure 6. Comparison of unconfined compressive strength of soil stabilized CMC-MMA emulsion polymer measured using dry and wet methods.

The comparison of unconfined compressive strength of soil stabilized CMC-MMA emulsion polymer measured using dry and wet methods is presented in Figure 6. The results indicated that the immersion of the soil samples in water for 3 minutes could significantly reduce the unconfined compressive strength of the soil. This shows that our material has lower durability under wet conditions. Water is likely to deteriorate the structure of the specimen and reduce the load-bearing capacity of the treated samples (Zandieh & Yasrobi, 2010).

#### pH of Polymer-stabilized Soil

The pH of the soil stabilized CMC-MMA emulsion polymer, and the untreated soil is shown in Figure 7. The results indicated that the pH of the soil was not greatly affected by the addition of a CMC-

MMA emulsion polymer. A slight increase in pH of the soil treated with the CMC-MMA emulsion polymer was due to the more basic nature of the polymer than the laterite soil. Soil stabilization employing the CMC-MMA emulsion polymer was not likely to change the acidity of the soil and thus would not cause any harm to the environment (Mishra, Bhatia, & S Nair, 2021).

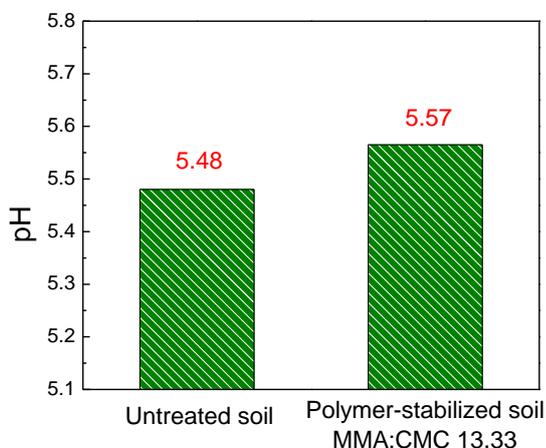


Figure 7. Comparison of pH of untreated soil vs. soil stabilized with MMA:CMC 13.33 emulsion polymer.

## CONCLUSION

In summary, CMC-MMA emulsion polymers were synthesized using different MMA to CMC mass ratios with SDS as the surfactant. The solids content in the prepared emulsion polymers were in the range of 5.52-17.02%. All variations produced high polymer yields (above 88.85%). Characterization of the polymers using FTIR spectroscopy indicated that the graft copolymerization was accomplished, with the tendency to undergo homopolymerization reactions with increased MMA fractions in the reactant. The viscosity of the emulsion polymers measured at room temperature was in the range of 1.2-3.2 cP. Unconfined compressive strength measurements of the polymer-stabilized soils showed a maximum compressive strength of 564 and 162 psi, respectively, obtained from the dry and wet methods. Besides, the rise of MMA fraction in the emulsion polymers tended to increase the compressive strength of the soil. A slight increase in soil pH was also noticed, indicating the basic nature of the materials. The graft CMC-MMA copolymers may find potential applications in the road construction industry that can replace asphalt pavements in the long run.

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