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Research Article

Dynamic mechanical properties of epoxy resin matrix reinforced with sugarcane bagasse

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ABSTRACT

The dynamic mechanical analysis was performed on five different formulations of a reinforced epoxy resin matrix with sugarcane bagasse (SCB) using hand lay-up technique. The viscoelastic behaviours of the composites were studied by analyzing the thermal parameters such as storage modulus E'(MPa), loss modulus E''(MPa), and $\tan\delta$ as the temperature increases at periodic stress frequencies of 2.5, 5, and 10 Hz using Dynamic Mechanical Analyzer (DMA). The results revealed that E'(MPa) and E''(MPa) decreased with increasing temperature for all composites, while the $\tan\delta$ increased with increasing temperatures. As the frequency of oscillation is increased, the viscoelastic parameters was seen to increase as well. However, the $\tan\delta$ (damping coefficient) was found to decrease with increase in SCB fibre loading due to the good load-bearing capacity of the composites. The $\tan\delta$ peak temperature of the composites was found to be higher than the onset temperature of E'(MPa) and peak temperature of E''(MPa) which depict higher accuracy. Futhermore, it was seen that the increase in SCB fibre content in the epoxy resin increased the glass transition temperature (T_g).

Keywords: Viscoelastic parameters, storage modulus, loss modulus, epoxy resin; dynamic mechanical properties.

Şeker kamışı küspesi ile güçlendirilmiş epoksi reçine matriksinin dinamik mekaniksel özellikleri

ÖZ

Dinamik mekanik analiz, el yatırma tekniği kullanılarak şeker kamışı küspesi (SCB) ile güçlendirilmiş epoksi reçine matrisinin beş farklı formülasyonu üzerinde gerçekleştirildi. Kompozitlerin viskoelastik davranışları, dinamik mekanik analiz cihazı (DMA) kullanılarak sıcaklık 2,5, 5 ve 10 Hz lik periyodik stres frekanslarında arttıkça depolama modülü E'(MPa), kayıp modülü E''(MPa) ve $\tan\delta$ gibi termal parametrelerin analiz edilmesiyle incelendi. Sonuçlar, E'(MPa) ve E''(MPa)' nın tüm kompozitler için artan sıcaklıkla azaldığını gösterdi, halbuki $\tan\delta$ ise artan sıcaklıkla artmıştır. Salınım frekansı arttıkça viskoelastik parametrelerin de arttığı görüldü. Bununla birlikte, $\tan\delta$' ın (sönümleme katsayısı) kompozitlerin iyi yük taşıma kapasitesi nedeniyle SCB fiber yükündeki artışla azaldığı bulundu. Kompozitlerin $\tan\delta$ pik sıcaklığının, daha yüksek doğruluğu gösteren E'(MPa) başlangıç sıcaklığından ve E''(MPa) pik sıcaklığından daha yüksek olduğu bulundu. Ayrıca epoksi reçinesindeki SCB içeriğindeki artışın ise camsı geçiş sıcaklığını (T_g) arttırdığı görüldü.

Anahtar Kelimeler: Viskoeleastik parametreler, depolama modülü, kayıp modül, epoksi reçine, dinamik mekanik özellikler.

1. INTRODUCTION

Nowadays, the natural-fibre reinforced polymeric materials have been utilized in many applications in a view of their easy availability, low cost, lightweight, high specific modulus, non-toxic and pollution-free production.[1] Due to the recent increase of environmental alertness, several studies were done on natural fibres as reinforcement or filler in composite so as to replace synthetic fibre.[2] Fundamentally, the use of natural fibre in polymers to form composite has numerous environmental benefits such as low pollutant, toxic chemical emissions, and increases dependence on non-renewable nergy ratio to material sources, enhanced energy recovery, low greenhouse gas emissions, and the end of life biodegradability of components. Such superior environmental performances are important driver of increased future use of natural fibre composites.[3] Advanced polymer composites are a relatively new set of materials that exhibit superior properties in comparison to traditional ones like steel and aluminum alloys in engineering applications.[3,4] In addition, natural fibres as reinforcement alternative in polymers have gained the attention of numerous material scientists because of their superior advantages over conventional carbon fibres and glass.[5,6] Dynamic thermomechanical analyzer (DTMA) is an instrument used for thermal analysis which measures the viscoelastic responses as the material deforms with temperature.[6] The viscoelastic response or parameter includes storage modulus (E'), loss modulus (E''), and modulus ratio (tan δ).[6] E' is defined as the stored energy per oscillating cycle which determines the elastic nature of the composite.[6] E'' is defined as the energy released per oscillating cycle which determines the viscous nature of the composite.[5,6] tan δ is the ratio between loss modulus to storage modulus which gives information about the viscoelastic nature or viscoelasticity of the composite material.[6] The oscillating frequency effects on the viscoelastic parameters of polymers were reported in many literatures, which gave information about the molecular motion on the polymer backbone.[6,7] Sugarcane bagasse fibres are environmental wastes that litter our environment. As such, utilizing the fibres as reinforcement or fillers in polymers could convert waste to wealth.[8] In our previous paper, we have reported the influence of the sugarcane bagasse powder addition on the viscoelastic parameters and activation energy of epoxy resin matrix at 10 Hz frequency only. However, glass transition temperature (T_g) and variation in frequencies on the viscoelastic parameters of the composites has not been reported.[9] Therefore, the ultimate goal of the present findings is to determine T_g, the effect of oscillation frequency on the viscoelastic parameters namely, E', E'', and tan δ as a function of the temperature of the SCB/Epoxy resin composite using dynamic mechanical analyzer (DMA).

2. EXPERIMENTAL

2.1. Methodology

The sample preparation method employed in this paper was adopted from our previous report.[9] Fresh sugarcane bagasse waste was sourced from a sugarcane juice center in Samaru Zaria of Kaduna State, Nigeria. The fresh bagasse was thoroughly washed with distilled water to remove sugar and dirt particles, then allowed to dried for two weeks. The dried sample was pulverized using a Lab milling equipment, and then sieved to 75 µm particle size using digital high-frequency sieve shaker. In addition, chemical pretreatment or fibre modification was not carried out in the present study. A bisphenol class of epoxy resin (3554A), and hardener (3554B) were supplied by a local supplier in Nigeria. In formulating the polymer composite, the hand lay-up method was adopted by manual stirring of the mixture with a rod, and the formulation of the SCB and epoxy resin were varied. The percent weight of reinforcement was weighed for the ratios of 0% wt (control), 10% wt, 20% wt, 30% wt, 40% wt and 50% wt of SCB/Epoxy while the epoxy matrix was formulated in the ratio of 2:1 (weight of epoxy resin to weight of hardener) as shown in Table 1.[9] The weighted matrix was mixed thoroughly with the SCB weight percentage at low speed for 15 min until the mixture became distributed evenly. Before demolding, petroleum jelly was applied to the surface of the mould with dimension (120 × 100 × 5 mm) before pouring the mixture into the mould. Thus, it prevents the composite from sticking to the mould and also aids the removal of the composite after curing. The composite was allowed to cure for 24 hours at temperature of 28°C before demoulding, then cut into suitable dimensions for further tests according to the ASTM standards.

Table 1. Nomenclature of the SCB/Epoxy resin composite materials.

Sample	Ratio	Matrix (g) 2:1		SCB (g)
		Epoxy resin	Hardener	
Pure Epoxy	100:0	66.60	33.30	0
10% wt SCB/Epoxy	10:90	60.00	30.00	10
20% wt SCB/Epoxy	20:80	53.24	26.76	20
30% wt SCB/Epoxy	30:70	46.60	23.40	30
40% wt SCB/Epoxy	40:60	40.00	20.00	40
50% wt SCB/Epoxy	50:50	33.30	16.70	50

2.2. Dynamic mechanical analyzer (DMA)

The DMA test was carried out using DMA 242E Artemis analyzer in the strength of materials laboratory, Mechanical Engineering Department, ABU Zaria in accordance with ASTM D7028 standard method.[10] The viscoelastic test parameters were initially configured via Proteus software using a personal computer. The viscoelastic response encompasses both elastic and viscous behavior of the composite. As such, the phase lag between applied oscillatory stress and the measured strain is between 0-90°. The applied stress (α), resultant strain (β), E', E'', and tan δ are given by the following equations.

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$$\alpha = \alpha_0 \sin(t\omega + \delta) \quad (1)$$

$$\beta = \beta_0 \sin(t\omega) \quad (2)$$

$$E' = \left(\frac{\alpha_0}{\beta_0}\right) \cos\delta \quad (3)$$

$$E'' = \left(\frac{\alpha_0}{\beta_0}\right) \sin\delta \quad (4)$$

$$\tan\delta = \left(\frac{E''}{E'}\right) \quad (5)$$

Where δ is the phase lag between stress and strain in MPa, ω is frequency of strain oscillation, α_0 is the stress amplitude, and β_0 is the strain amplitude.

However, the instrument was configured at furnace temperature range (30 to 120 degree Celsius), dynamic load/force (2.18 N), test frequencies (2.5, 5, and 10 Hz), and heating rate (5K/min) respectively. The sample with 40 x 12 x 5 mm dimension was loaded on to the machine using the three-point bending sample holder, then locked into the furnace to start the analysis. The rationale behind using the furnace temperatures of 30-120°C is to fully monitor the temperature scan as the materials deforms from glassy region at room temperature (lower temperature) to a rubbery region (higher temperature) where the material completely deforms. In addition, the T_g is expected to fall within this temperature range.

3. RESULTS AND DISCUSSION

3.1. DMA test curves and glass transition temperature (T_g)

DMA describes the stiffness stability of the composite material as a function of temperature when subjected to dynamic loading, and the modulus of materials is a measure of its stiffness property.[9,10] Figure 1 illustrates viscoelastic parameters such as E', E'', and tan δ of neat epoxy resin (control) with increasing temperature at oscillation frequencies of 2.5, 5, and 10 Hz respectively. The E' (MPa) curve shows the stability of the neat matrix under dynamic loading with an onset temperature of 35.6°C and inflection point of 38.8°C at 10 Hz. The E''(MPa) curve depicts peak maximum at 31.2°C (193 MPa) while tan δ curve describes the viscoelasticity of a neat epoxy resin matrix with a peak temperature of 63.2°C (1.056) at 10 Hz.

In **Figure 2**, the E'(MPa) curve displays an increased onset temperature of 38.0°C and inflection temperature of 41.7°C due to the incorporation of 10% wt of SCB into the matrix. The E''(MPa) curve also shows an increased onset temperature of 36.0°C (57 MPa), while tan δ curve indicates a decrease in peak temperature of 59.9°C (1.018).

Figure 3 shows better thermal stability of the 20% wt SCB/Epoxy composite under dynamic loading with an onset temperature of 45.60°C on E' curve at 10 Hz when compared to the neat epoxy resin and 10% wt SCB reinforcements respectively. The E'' curve depicts an increase in energy dissipation at peak maximum of 41.0°C (118 MPa), while the tan δ curve reveals an increased peak temperature of 66.5°C (0.716) compared to neat epoxy.

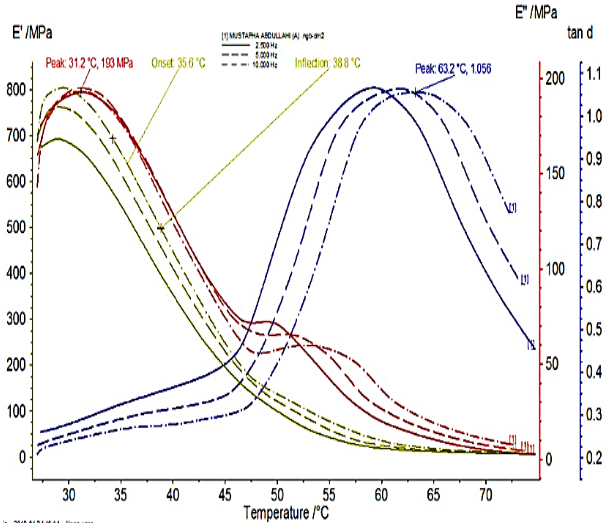

Figure 1. DMA test curves for 0% wt SCB/Epoxy resin composite at oscillation frequencies of 2.5, 5, and 10 Hz (neat epoxy).

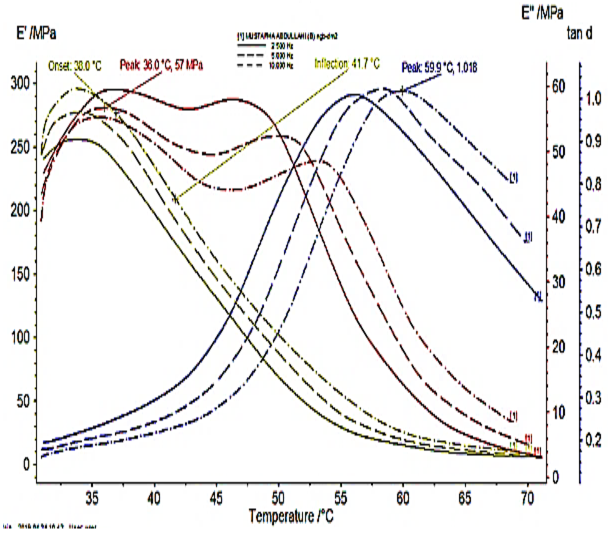

Figure 2. DMA test curves for 10% wt SCB/Epoxy resin composite at oscillation frequencies of 2.5, 5, and 10 Hz.

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The *E'* curve of 30% wt SCB/Epoxy resin composite reveals similar thermal stability compared to 20% wt SCB reinforcement with an onset temperature of 44.0°C. Also, the peak maximum at 40.6°C and 158 MPa of loss modulus was observed, while the viscoelastic nature is eminent at tan δ value of 0.689 and peak temperature of 66.7°C as shown in Figure 4.

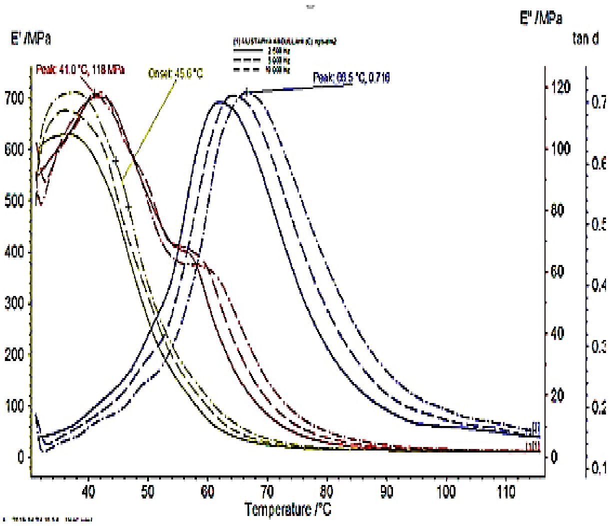

Figure 3. DMA test curves for 20% wt SCB/Epoxy resin composite at oscillation frequencies of 2.5, 5, and 10 Hz.

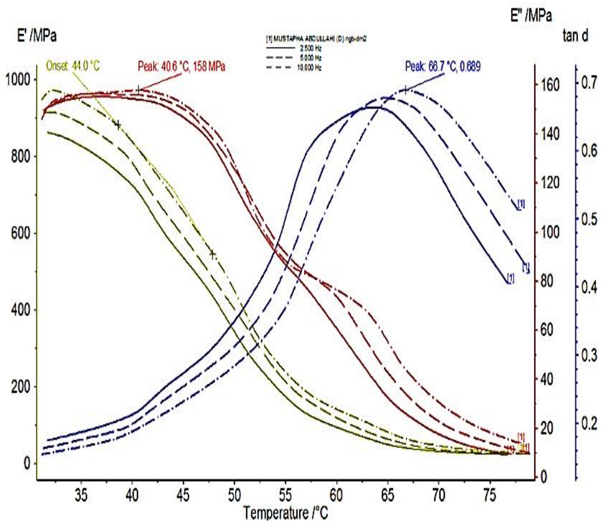

Figure 4. DMA test curves for 30% wt SCB/Epoxy resin composite at oscillation frequencies of 2.,5, 5, and 10 Hz.

Similar improvement in thermal stability is observed in *E'* curve for 40% wt SCB under dynamic loading at an onset temperature of 43.0°C and inflection point of 46.0°C respectively. The peak maximum of *E''* was observed at 36.3°C (169 MPa) and the viscoelastic nature of the composite is imminent with a loss factor of 0.683 and peak temperature (67.8°C) as shown in Figure 5.

In Figure 6, the *E'* curve shows that the 50% wt SCB/Epoxy composite under dynamic loading before the onset temperature (54.1°C) and inflection point (60.3°C) at 10 Hz.

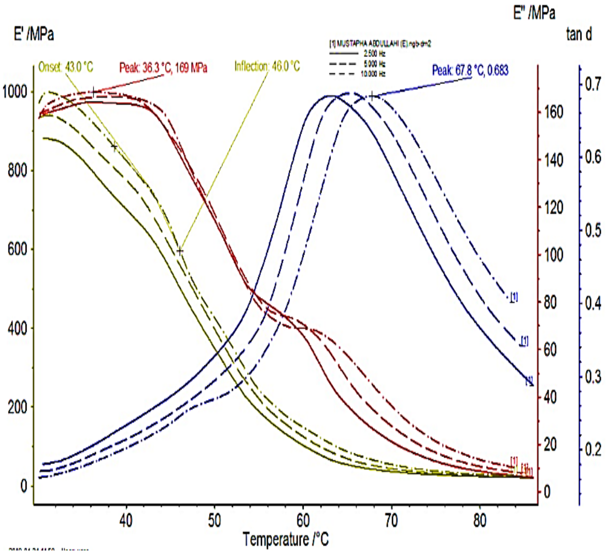

Figure 5. DMA test curves for 40% wt SCB/Epoxy resin composite at oscillation frequencies of 2.5, 5, and 10 Hz.

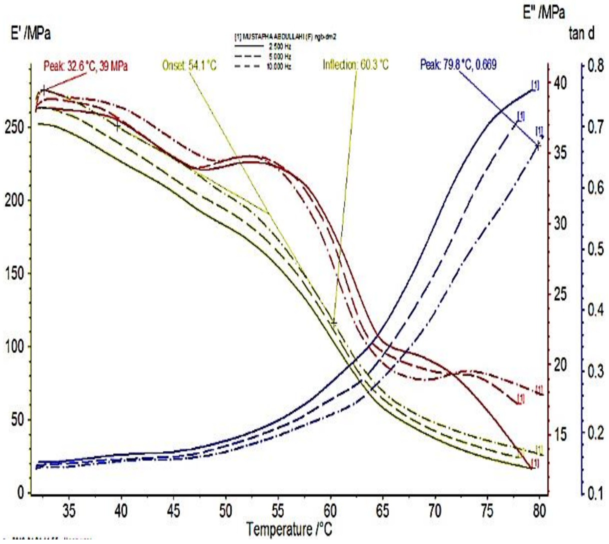

Figure 6. DMA test curves for 50% wt SCB/Epoxy resin composite at oscillation frequencies of 2.5, 5, and 10 Hz.

The *E*" curve depicts a peak maximum at 32.6°C and 39 MPa. However, the viscoelastic nature of the composite is eminent at the tan δ value of 0.669 and peak temperature of 79.8°C.

From the above DMA curves (Figures 1-6), it can be observed that E'(MPa) and $E"$(MPa) curves decrease with an increase in temperature for all composites, while the $\tan\delta$ curve increases with an increase in temperatures up to a particular peak height before decreasing. The steep decrease in E'(MPa) and $E"$(MPa) curves depict the glass transition region which is attributed to the enhancement of molecular motion with gain of energy.[11] Also, it can be seen that the viscoelastic parameters increase with an increase in the oscillation frequencies. The T_g is defined as the temperature region at which composite material changes from a glassy, “rigid” to a rubbery state.[13,14] In physical chemistry of polymers, the T_g region is the region where the polymer chains gains sufficient energy to increase the molecular mobility or motion in the matrix which resulted a sudden change in its physical and mechanical properties.[15] The T_g computed by storage modulus (E'), loss modulus (E'') and tan δ curves for the SCB reinforced epoxy composites are not alike. The tan δ peak temperatures in this study are found to be higher than the peak temperatures obtained from E'(MPa) and $E"$(MPa) respectively. In essence, the T_g is recorded as the onset temperature for E'(MPa) curve, peak temperature for $E"(MPa)$ and $\tan\delta$ curve accordingly. The summary of the recorded glass transition temperatures for the SCB reinforced epoxy resin composites obtained from the DMA test curves at 10 Hz is shown in Table 2. It is deduced that increase in the SCB fibre content increases the glass transition temperatures. In addition, the higher values of the T_g obtained from the tan δ curve depicts higher accuracy.[16] The results show the increase in E'(MPa) and $E"$(MPa) values with an increase in SCB fibre content in the epoxy matrix due to good interfacial adhesion between the fibre and the matrix which increased its thermal stability.[6,9]

3.2. Viscoelastic parameters at different oscillating frequencies

The viscoelastic parameters of composites are the E', E'', and tan δ. The storage modulus of a polymer composite material describes how materials are stiffer.[12] The loss modulus of a polymer composite material is defined as the highest energy dissipated by the composite material during deformation, and it is the viscous response which depends upon the motion of polymeric molecules in the composite.[12] tan δ coefficient or loss factor of a polymer composite gives balance information between the elastic and viscous phases in a polymeric structure.[10,12] The variation in oscillation frequencies (2.5, 5, and 10 Hz) of the viscoelastic parameters in the glass transition region is reported in Table 3. From the results, it is deduced that the viscoelastic parameters increase with an increase in the frequency of oscillation. This may be attributed to the increase in number of dynamic cycles which resulted more rapid or faster molecular motion of the polymer chain thereby increasing the viscoelastic parameters.[10]

Table 2. Peak temperatures for SCB/Epoxy resin composites

Composites	**T_g (°C) from E' Onset peak (10 Hz)**	**T_g (°C) from E'' peak (10 Hz)**	**T_g (°C) from $\tan\delta$ peak (10 Hz)**
0% wt SCB/Epoxy	35.6	31.2	63.2
10% wt SCB/Epoxy	38.0	36.0	59.9
20% wt SCB/Epoxy	45.6	41.6	66.5
30% wt SCB/Epoxy	44.0	40.6	66.7
40% wt SCB/Epoxy	43.0	36.3	67.8
50% wt SCB/Epoxy	54.1	32.6	79.8

Table 3. Viscoelastic parameters at glass transition peak heights

Composites	**Frequency (Hz)**	**E'(MPa)**	**$E"$(MPa)**	**tan δ**
0% wt SCB/Epoxy	2.5	547.876	192.624	0.9949
	5	617.238	193.031	1.05731
	10	663.154	193.660	1.0560
10% wt SCB/Epoxy	2.5	226.263	55.3441	0.92433
	5	250.042	56.8887	0.99475
	10	265.18	57.0000	1.0180
20% wt SCB/Epoxy	2.5	456.291	117.160	0.65640
	5	487.146	117.818	0.70335
	10	550.016	118.000	0.7180
30% wt SCB/Epoxy	2.5	564.515	154.170	0.64665
	5	620.71	155.800	0.67454
	10	687.712	158.000	0.68900
40% wt SCB/Epoxy	2.5	626.772	164.133	0.63127
	5	690.128	166.409	0.66776
	10	744.173	169.000	0.6830
50% wt SCB/Epoxy	2.5	155.751	38.2076	0.62111
	5	165.982	38.8292	0.65721
	10	174.522	39.0000	0.6690

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4. CONCLUSIONS

The dynamic mechanical analysis is carried out successfully on five different formulations of SCB/Epoxy resin composites at different oscillation frequencies (2.5, 5, and 10 Hz) and the following conclusions are attained.

- The viscoelastic parameters (E', E'' and $\tan\delta$) increase with an increase in oscillation frequencies.
- The storage modulus E'(MPa) and loss modulus E''(MPa) decrease with an increase in temperature for all composites, while the $\tan\delta$ increases with the increases in temperatures up to the highest peak before decreased.
- The peak temperature of $\tan\ \delta$ of the composites are higher than the onset temperature from E'(MPa) and peak temperature of E''(MPa). However, an increase in SCB fibre content increases the T_g of the composites.

- The acceptable dynamic mechanical properties of these optimum composites reveals that they can have end-use application in making the laminated circuit board and electronic component encapsulation.

Conflict of interests

Author declares that there is no a conflict of interest with any person, institute, company, etc.

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