Effect of Carbon Black Type and Loading on Properties of Natural Rubber/Butadiene Rubber Blends for Solid Tire Tread Compounds

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Abstract- The paper investigates the effect of carbon black (CB) types and loading on curing and physico-mechanical properties of solid tire tread compounds comprised of Natural Rubber (NR) and Butadiene Rubber (BR) blend. N326, N330, and N375 were the CB types loaded in the rubber compounds at 53-60 parts per hundred parts of rubber (phr), which was a predetermined loading level. Tensile strength, tear strength, hardness, abrasion resistance, and cure characteristics were tested, keeping the N330 CB type at 60 phr loading as the control compound. The cure characteristics of all the compounds showed good processability, processing safety, and curing efficiency. The CB type N375 at 55 phr loading showed better tear strength, tensile properties, hardness, and abrasion resistance than the other compounds. Of the three types of CBs used in tire tread compounds, the N375 CB type was the ideal type for solid tire treads, according to this study. However, there is a choice of CB type and loading level as the requirement of mechanical properties.

Index Terms- Butadiene Rubber, Carbon Black, Natural Rubber, Blends, Solid Tire Tread

I. INTRODUCTION

In terms of tremendous development throughout the last decade, the tire business has always come out on top, owing to the amount of research on rubber. These studies have been devoted to tire tread because this is the section of the tire that has the most impact during use. Natural Rubber is commonly used in all types of tires, particularly in heavy truck tires and aircraft tires, to achieve a lower heat build-up and mechanical strength. Furthermore, various components of passenger car tires, such as the bead filler, sidewall, and tread compounds, are made of NR because of NR's lower heat built-up, flexing, fatigue, and mechanical properties (Sattayanurak *et al*, 2020). Tires are made up of various components, each containing about 30% particulate fillers like carbon black and silica (Brinke *et al*., 2003). Hence, they are considered important additives in rubber compounding (Wijesinghe *et al*., 2016). Carbon black was the first reinforcing filler used in tire compounds to improve modulus, tensile, and tear strength, and skid resistance (Kim and Jeong, 2005: Poikelispää *et al*., 2013: Wijesinghe *et al*., 2022: Wijesinghe *et al*., 2021) (Chauhan & Chawla, 2016). Robertson *et al*. (2019) have mentioned that the strength of rubber depends on its chemical structure and viscoelastic behavior. Numerous studies have been done on the mechanical and dynamic properties of CB-filled tire compounds. The particle size and surface area of CB have been identified as essential parameters for increasing tire performance (Zafarmehrabian *et al*., 2012). It has been mentioned that carbon black with small particle sizes is used in the tire tread, where wear resistance is an important parameter (Ishikawa, 2011). Atashi & Shiva, (2010) investigated the optimization of failures of passenger tire tread compounds made up of Styrene Butadiene Rubber (SBR), Natural Rubber, and Butadiene Rubber by varying curing conditions and modeling the rubber compound's performance. According to the studies conducted by Pal *et al*., (2010) on NR/BR/high styrene rubber (HSR) compound, varying carbon black contents showed an increased abrasion resistance and additionally, observed an increase in the strength of the compound when particle size reduced to the nanoscale. Farida *et al*., (2019) have mentioned that the addition of carbon black to natural rubber/polypropylene compounds can increase the tensile properties while decreasing the elongation at break.

The specific surface area and structure of carbon black influence the level of entanglement with polymer molecules, affecting the reinforcement (Salaeh & Nakason, 2012). The high specific surface area (60-90 m^2g^{-1}) of CB primary particles can spontaneously form nanostructured aggregates and agglomerated to result in distinct porous architectures based on nanometric spherical particles (Li *et al*., 2008). Studies have been conducted using different types of carbon black to understand the effect of CB structure on the properties of composites. Therefore, different CB types can show different structural arrangements in the polymer composite due to intrinsic particle properties, such as size, shape, etc. A study conducted using different carbon black-filled ethylene propylene diene monomers (EPDM) rubber showed higher mechanical and electric properties for N472 than the other black types used [others being N330, N550, N770, and SCB (Spraying Carbon Black)] (Li *et al*., 2008). According to the literature, the capacity of CB to aggregate into particles with precise nano-size geometries determines the polymer-filler interactions of polymer composites (Hjelm *et al*., 1994). Several agglomerated 3D structures of different CB types in polymers have been investigated before. Kwon *et al*., (2014) have reported the agglomeration of N326 CB type in polyimide composites. Quan *et al*., (2018) have reported the formation of grape-like aggregated structures of microscopically spherical particles of N330 CB type in elastometic matrices that affect the reinforcement. Also, they have observed countless pores within these cluster aggregates with no obvious boundaries. The N375 CB type has shown spontaneous aggregates due to high surface area (Mazzaracchio *et al*., 2019). To our knowledge, apart from a few studies, there has not been much research on the influence of carbon black type and loading level on the mechanical properties of solid tire tread compounds made of natural rubber and butadiene rubber blends (NR/BR blends) (Chung *et al*., 2002: Wang *et al*., 2001). Therefore, to understand the effect of CB type and loading level on the curing and physico-mechanical properties of NR/BR blends for solid tire tread compounds, three different CB types (N326, N330, and N375) with average particle sizes of 28-36 nm at different loading levels were incorporated into such blends in this study followed by evaluating the curing and physico-mechanical properties.

II. MATERIALS AND METHODS

Preparation of compounds

A basic compounding formulation was developed using an industrial formulation for the solid tire tread compounds. The compound was prepared by blending natural rubber and butadiene rubber with other compounding ingredients, as mentioned in **Table 1**.

Table 1: The formulation for tire tread compounds

Ingredients	Amount in parts per hundred parts of rubber (phr)
Natural Rubber	60
Butadiene Rubber	40
Carbon black (N329, N330, N375)	53, 55, 57, 60
ZnO	4.5
Stearic acid	2.5
IPPD	2
TMQ	1
Sulfur	1.2
TBBS	1
PVI	0.25

Note: Zinc oxide-(ZnO), N-Isopropyl-N′-phenyl-1,4-phenylenediamine (IPPD), 2,2,4-trimethyl-1,2-dihydroquinoline-(TMQ),N-tetrabutyl-2-benzothiazylsulfinamide-(TBBS), Pre-Vulcanizing Inhibitor-(PVI)

The sulfur, N-tetrabutyl-2-benzothiazylsulfinamide (TBBS) levels in the compounding formulation were fine-tuned by pilot studies. Later, the CB level of each type of CB (N326, N330, and N375) in the compounds was varied at 53, 55, 57, and 60 phr. The N330 CB type at 60 phr loading level was the control sample (N330@60) of the study as it is widely used in the tire industry. The mixing method of compounds is demonstrated in **Table 2**. The first stage of mixing the compounds was carried out using a Laboratory Banbury (Techno Lab BR1600) at 70 ºC. In this stage, the viscosity of rubbers was reduced via mastication, and CBs were incorporated into rubber blends, achieving a good dispersion. After mixing these materials with rubber, the initial compounds were dumped and matured at room temperature (25 °C) for 16 hours. In the second mixing stage, sulfur and accelerators were mixed with the matured compounds of the first stage using a two-roll mill at below 100 °C.

Evaluation of cure characteristics

Rheographs of compounds were obtained using an Oscillating Disk Rheometer (2000 M/S Alpha technologies, USA) at 150ºC following the ISO 3417:2008 procedure. The scorch time (Ts_2), optimum cure time (Tc_{90}), cure rate index (CRI), maximum torque (M_H), minimum torque (M_L), and delta cure ($M_{H-} M_L$) of compounds were derived from the rheographs.

Table 2: Mixing cycle of the compounds

Mixing stage	Ingredient	Time (seconds ±20)	Mixing temperature (°C)
First stage	NR	0th	70 ± 5
	BR	60th	
	ZnO, stearic acid, IPPD	120th	
	TMQ	300th	
	Carbon black	360th	
	Dump	540th	
Second stage	NR/BR blend	0th	<100
	TBBS	60th	
	Sulfur	120th	
	Dump	180th	

Evaluation of physico-mechanical properties

The test samples were prepared using the hydraulic press machine (Yeji Corporation, Taiwan), keeping the temperature and pressure at 150 ºC and 20 MPa, respectively. The optimum cure times (T_{c90}) of each compound obtained from the rheographs were used to cure the samples during molding. After curing the test samples, they were cooled with water to prevent further vulcanization. Then, the samples were conditioned at room temperature for 16 hours before performing the physico-mechanical tests. The tensile properties were determined by the tensile testing machine (Gotech Testing Machines Inc) at a crosshead speed of 500 millimeters per minute ($mmmin^{-1}$) according to ISO 37:2017 standard using dumb-bell shaped specimens.

The tear strength of vulcanizates was determined by the same tensile testing machine at a crosshead speed of 500 mmmin^{-1} using angular test pieces following the ISO 34-1:2015 procedure. A manual thickness gauge was used to determine the thickness of the test pieces. The hardness of the samples was measured using a mechanical hardness tester according to ISO 48-2:2018 procedure. The abrasion volume loss was determined using a DIN abrasion tester (Rotary Drum Abrader) following the ASTM D5963 test method. Finally, statistical analysis was done using Minitab 18 statistical software.

III. RESULTS AND DISCUSSION

Cure characteristics

The cure characteristics of compounds are shown in **Table 3**. The lowest torque (M_L) indicates the stock viscosity of rubber compounds, and that of our compounds has varied between 14 and 19 dNm. A slight increment of M_L can be observed when the CB content was increased from 53 to 60 phr. The maximum torque (M_H) values depend on the crosslinking density and chain entanglements; thus, it is related to the fact that CB alters the crosslink density by reacting with other chemical ingredients, which leads to a higher torque (Yasir *et al*., 2015). In our compounds, the M_H values have decreased with the type of CB, which may be due to the variation in particle size. Delta cure (M_H-M_L) is the parameter that indicates the degree of chemical crosslinking (Rattanasom & Prasertsri, 2009). It is reported that the reaction between rubber and the curing system can affect the crosslinking process (Pöschl *et al*., 2020), whilst Rattanasom & Prasertsri, (2009) state that smaller particle size can be a reason for having higher crosslinking density. The delta cure values of N330 are comparatively higher than other compounds. This can be related to the structure of the N330 CB type. The scorch time (T_{S2}) provides information regarding processing safety, and compounds with N375 CB type have shown a higher value compared to other CB types. The optimum cure time (T_{C90}) of the compounds has shown a slight variation compared to the control. Further, T_{S2} and T_{C90} values have decreased with the increase in CB loading.

Table 3: Cure characteristics of the compounds

CB composition	M_L (dNm)	M_H (dNm)	Delta cure (dNm)	T_{S2} (min)	T_{C90} (min)	C.R.I. (min^{-1})
N326@53	16.7	87.4	70.7	6.08	14.0	12.5
N326@55	18.1	85.9	67.3	6.15	14.5	11.9
N326@57	18.2	88.7	70.5	5.49	13.1	12.9
N326 @60	18.3	89.0	70.7	6.2	13.5	13.6
N330@53	15.4	85.4	70.0	6.54	14.5	12.5
N330@55	14.3	83.0	68.7	6.45	14.2	12.8
N330@57	15.7	83.4	67.6	6.28	13.5	13.7
***N330@60**	**16.8**	**84.3**	**67.5**	**6.22**	**14.1**	**12.5**
N375@53	15.8	77.8	62.0	8.05	17.3	10.7
N375@55	16.6	79.1	62.5	7.48	16.4	11.2
N375@57	17.1	80.1	62.9	7.26	16.1	11.2

The cure rate index (CRI) of compounds has varied between 10-13 min^{-1}, where a slight reduction can be observed in compounds with N375 CB type. This may be attributed to the effect of CB types on the curing process (Saad *et al*., 2009).

Physico-mechanical properties

The variation of the tear strength of NR/BR blends with CB composition is shown in **Figure 1.** According to the theory, tear strength measures the resistance of a rubber compound to initiate and propagate a crack **(**Ciesielski, 1999). The tear strength of NR/BR blend compounds with each CB type has increased with the filler CB loading. The highest tear strength (130-145 Nmm^{-1}) was recorded by the compound containing the N375 CB type, except for the N375@53 compound with a tear strength of ca. 110 Nmm^{-1}. The CB composition of N326@57, N326@60, N330@55 are not statistically significant compared to the control (ca. 120 Nmm^{-1}), while that of other N326 and N330 CB types containing NR/BR blends are significantly lower (ca.100-115 Nmm^{-1}) to the control. The good dispersion of CB in rubber may be responsible for the formation of a physical barrier against crack propagation, resulting in increased tear strength (Abdelsalam *et al*., 2019; Mazzaracchio *et al*., 2019).

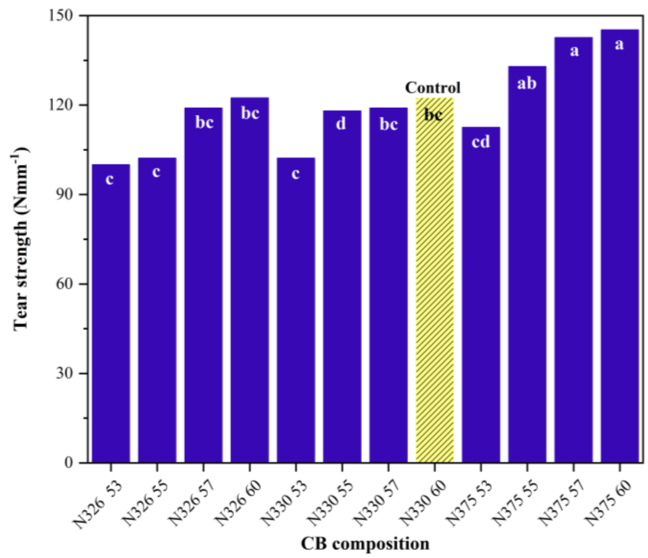

Figure 1: Tear strength of NR/BR blend compounds with the CB composition

Figure 2 shows the variation of tensile strength of NR/BR blends with CB composition. The control (N330@60) has shown about 220 kPa of tensile strength. The tensile strength of the N326 CB type containing blend compounds has varied from 210-230 kPa, which is similar to the N330 CB type containing blend compounds. However, NR/BR blends with N375 CB type have shown the highest tensile strength (ca. 235-255 kPa). Also, the tensile strength of all the types of CBs has increased up to the loading level of 55 phr and has reported a marked decline thereafter. According to the literature, the rubber-filler interactions can be attributed to the improvement of tensile strength (Pornprasit *et al*., 2016). Also, the agglomeration of CB particles at high loading levels could be attributed to the decline of strength at higher loading levels (Farida *et al*., 2019; Abdelsalam *et al*., 2019). Here, we have observed that there is a possibility of increasing the tensile strength with lower loading than the use of a 60 phr loading level.

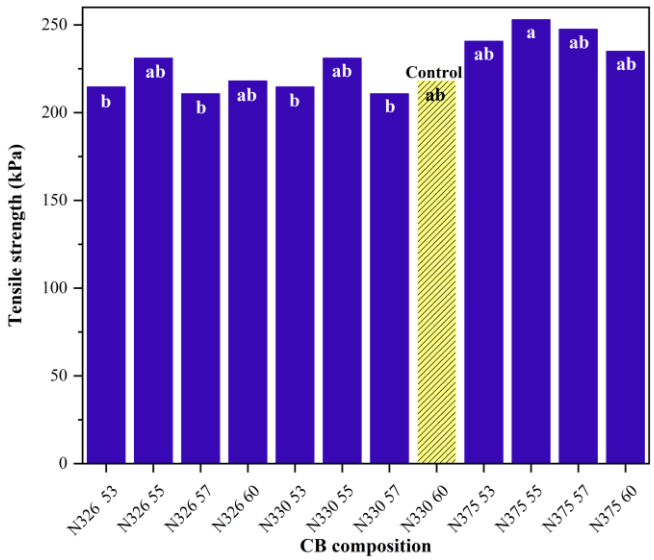

Figure 2: Tensile strength of NR/BR blend compounds with the CB composition

The variation of modulus of NR/BR blends with CB composition is shown in **Figure 3**. Accordingly, an increase in modulus can be observed when the CB type varies from N326 to N375. The CB type N375 has shown a higher modulus (ca. 130-150 kPa) in NR/BR blend compounds, which is greater than that of the control compound (ca. 110 kPa). Further, the modulus increases when the CB loading level varies from 53 to 60 phr. The lower modulus and tensile strength given by the N326 CB types can be attributed to the poor agglomerated structure of N326 in rubber compounds (Martínez *et al*., 2013). However, the improved modulus by other types of CBs can be assigned to the interactions between rubber and filler (Saad *et al*., 2009).

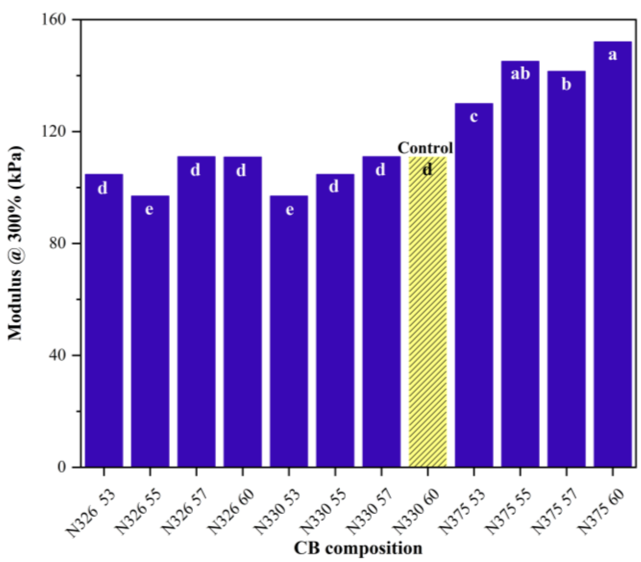

Figure 3: Modulus at 300% of NR/BR blend compounds with the CB composition

The variation of elongation at break NR/BR blends with CB composition is shown in **Figure 4.** The elongation at break of control is about 460%. Also, elongation at break of N326 and N330 CB types has varied between ca. 517-460%, while that of N375 CB type has varied between 470 and 417% with respect to the CB loading. The observed reduction of elongation at break of these compounds can be attributed to the reduction of the elasticity due to increased CB loading (Ismail *et al*., 2018). Compared to other CB types, N375 CB types in NR/BR blend compounds have shown a reduced elongation at break, which might be due to the improved rubber-filler interactions and aggregated structures formed in the blends that reinforce the compound (Mazzaracchio *et al*., 2019).

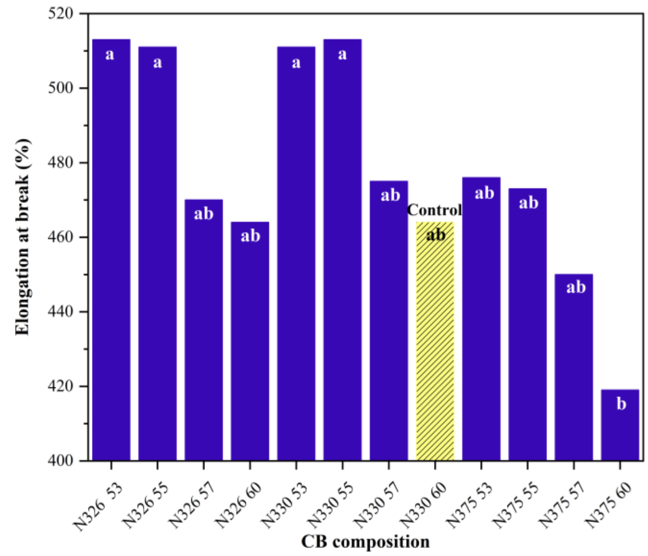

Figure 4: Elongation at break of NR/BR blend compounds with the CB composition

Figure 5 shows the variation in the hardness of NR/BR blends with CB composition. The hardness of rubber compounds is a measure of resistance to the deformation of rubber for indentation (Bijarimi *et al*., 2010). The results show that all the compounds have a hardness between 60-70 IRHD. The control has shown a hardness of 67 IRHD. The hardness of NR/BR blends with N326 CB types has varied between 62 and 67 IRHD, while a similar variation has been shown by N330 CB type containing blends when the loading level is varied from 53-60 phr. However, the blends with CB type N375 have shown the highest hardness values, which varied from 67-69 IRHD with the CB loading level. The improved aggregated structure and good dispersion of N375 in these blends can be attributed to the improved hardness (Mazzaracchio *et al*., 2019). Also, the increment of hardness with CB loading is similar to a previous study done with CB-filled acrylonitrile butadiene rubber (NBR) and recycled NBR blends. According to that study, the increased rigidity of rubber vulcanizates due to increased CB content can be attributed to the hardness increment (Husnan *et al*., 2018).

Figure 6 shows the variation of abrasion of NR/BR blends with CB composition. Abrasion resistance measures the ability of a material to withstand mechanical action, such as rubbing or scraping, which are critical in tire treads. The abrasion volume loss was used in this study to predict the resistance to wear of these NR/BR blend compounds. According to the figure, the abrasion volume loss of N326 and N330 CB type-containing blends has shown statistically non-significant values varied between 100-115 mm^3. Although these values satisfy the industrial requirement of abrasion volume loss, N375 CB type-containing blends have shown a remarkable reduction in abrasion volume loss (71-75 mm^3), indicating an improvement in abrasion resistance. In literature, it has been mentioned that when the structure of carbon black increases, the abrasion resistance also increases (Hong *et al*., 2007). Thus, it can be reported that the N375 CB type has a better structure that can improve mechanical properties and abrasion resistance of NR/NBR blend compounds for solid tire treads than the other CB types considered in this study.

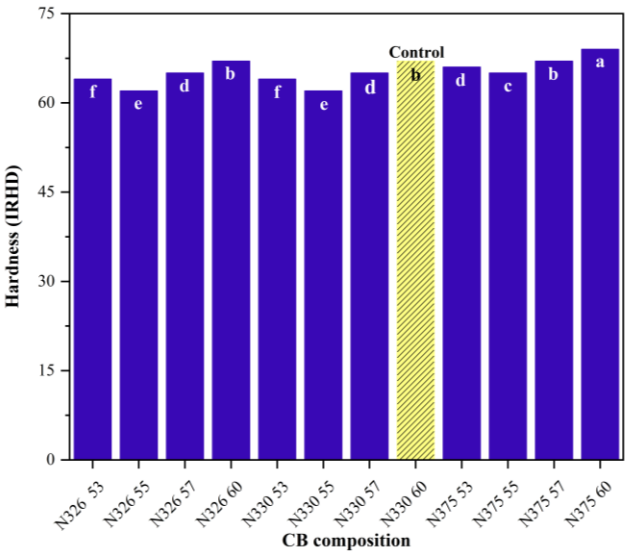

Figure 5: Hardness of NR/BR blend compounds with the CB composition

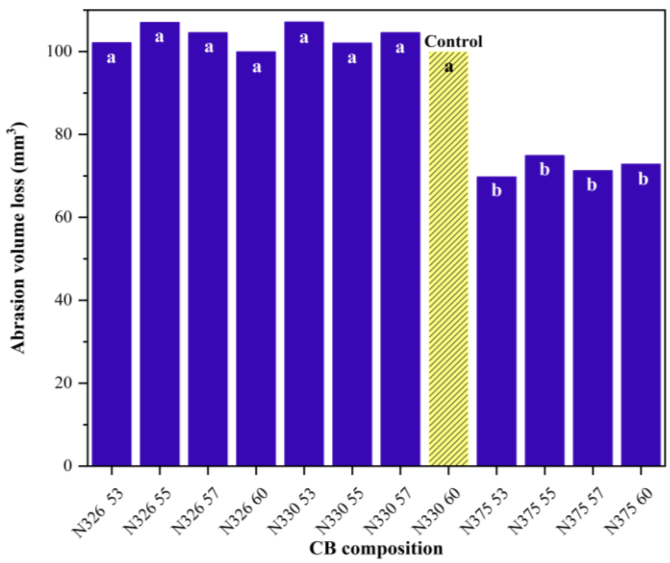

Figure 6: Abrasion volume loss of NR/BR blend compounds with the CB composition

IV. CONCLUSION

Three types of CBs (N326, N330, and N375) were incorporated into NR/BR blends for solid tire tread compounds varying the CB loading level at 53, 55, 57, and 60 phr. The cure characteristics of the blend compounds showed good processing safety and curing efficiency. The highest tear strength, tensile properties, hardness, and abrasion resistance were demonstrated by the N375 CB type containing NR/BR blends compared to the N326 and N330 CB types containing NR/BR blends. Overall, the properties of the N375 CB type have shown better improvement in physico-mechanical properties compared to other CB types at the loading levels of 53-60 phr. Based on the results, it can be concluded that there is a choice of CB type and loading level based on the required physico-mechanical properties of solid tire tread compounds.

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