

## Formulation of the Trae Product by Viscosity Modification with Dilution of Rae Material Utilizing Base Lubricating Oil

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### ABSTRACT

*The growing global demand for environmentally compliant materials has spurred safer alternatives to hazardous compounds in rubber processing oils. Regulations like the EU REACH Directive and Directive 2005/69/EC impose strict limits on polycyclic aromatic compounds (PCA) and polycyclic aromatic hydrocarbons (PAHs) in extender oils for tires and rubber, requiring innovative formulations. This study examines the Formulation of the Trae Product by Viscosity Modification with Dilution of Rae Material Utilizing Base Lubricating Oil by diluting residual aromatic extract (RAE) with base lubricating oil (LBO) to optimize rheology and compliance. Blending occurred at 100 °C for 60 minutes using RAE:LBO ratios of 85:15, 70:30, and 60:40. Results showed dilution reduced RAE kinematic viscosity from 18,487 mm<sup>2</sup>/s to 799.0 mm<sup>2</sup>/s at 40 °C and from 115.07 mm<sup>2</sup>/s to 31.71 mm<sup>2</sup>/s at 100 °C. PCA dropped from 14.1 wt% to below 3 wt%, with total PAH and benzo[a]pyrene (BaP) meeting EU Directive 2005/69/EC and REACH Annex XVII limits (<10 mg/kg and <1 mg/kg). The 70:30 ratio matched commercial TRAE products (H&R Vivatec 700 and 800) in viscosity with better environmental profile. This approach provides a sustainable path for low-PAH TRAE production.*

**Keywords:**

*viscosity modification; dilution method; Residual Aromatic Extract (RAE); base lubricating oil; PCA; PAH*

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### INTRODUCTION

The global industrial sector faces mounting pressure to reduce hazardous chemical content in petroleum-derived products, particularly in rubber processing applications. International regulatory frameworks, including the EU REACH Regulation (EC No. 1907/2006) and Directive 2005/69/EC, have established stringent limits on polycyclic aromatic hydrocarbons (PAHs) and polycyclic aromatic compounds (PCAs) in extender oils used in tire manufacturing and other rubber products. These regulations mandate that materials containing PCA levels exceeding 3% by weight must be classified as carcinogenic, mutagenic, or reprotoxic (CMR) substances, significantly restricting their commercial application. Furthermore, the directive specifies maximum allowable concentrations of 1 mg/kg for benzo[a]pyrene (BaP) and 10 mg/kg for the sum of eight priority PAHs, reflecting the severe health risks associated with long-term exposure to these compounds (European Parliament & Council, 2005; ECHA, 2025). This regulatory landscape has intensified the need for innovative processing technologies that can reduce aromatic content while maintaining the functional properties essential for industrial applications.

The oil and gas industry produces a wide range of products that are categorized into fuel oil (BBM) and non-fuel, each with technical specifications tailored to its characteristics and usage needs. that produces light fractions such as straight-run gasoline,

kerosene, and light Products can be produced through various stages of petroleum processing, ranging from primary processing gas oil, to vacuum residue as the basic material for non-fuel products such as lubricants and asphalt; secondary processing that includes cracking and reforming to produce reformat, cracked gasoline, and diesel; and tertiary processing that focuses on refining and blending to produce final products such as in the manufacture of avtur and rubber processing oil (RPO) (Speight, 2014).

Rubber processing oil (RPO) is a non-fuel product that is an important component in the rubber industry to improve the processability, flexibility, and mechanical properties of rubber compounds (Kaimai et al., 2001). RPO functions as a softener and extender in rubber mixing, so that it can reduce viscosity, increase elongation, and lower the glass transition temperature ( $T_g$ ) in rubber compounds (Bandyopadhyay, 2024; Sisanth et al., 2017). There are various types of rubber processing oil (RPO), namely aromatic oil, naphthenic oil, and paraffinic oil, each of which has its own characteristics and level of compatibility with rubber polymers (Chokanandsombat and Sirisinha, 2014).

One of the widely used RPO products is residual aromatic extract (RAE), which can be obtained from the fraction of petroleum residues and is known to have high viscosity and significant aromatic compound content. Residual aromatic extract (RAE) is designed to improve the processing properties of rubber compounds and plays a role in the manufacturing industry of vehicle tires, shoe soles, and a variety of high-quality parts. This makes RAE the right product choice for its application in the manufacturing industry.

Although rubber aromatic extract (RAE) products have been applied in various industrial processes, it should be noted that these products still have high viscosity values. Too high viscosity values can cause problems in the mixing process with rubber materials, which in turn can hinder production efficiency and affect the quality of the final product. In addition, RAE products also contain polycyclic aromatic hydrocarbons (PAHs). Polycyclic aromatic hydrocarbons (PAHs) are aromatic compounds that consist of two or more benzene rings fused in various structural configurations and contain no heteroatoms or substituent groups (Lawal, 2017). The presence of PAHs can cause carcinogenic, mutagenic, and immunosuppressive effects, threatening human health and damaging the environment (Shafy and Mansour, 2016; Patel et al., 2020).

Polycyclic aromatic compounds (PCAs) are a class of aromatic compounds that consist of two or more benzene rings joined together, encompassing both pure hydrocarbon compounds as well as heterocyclic and alkylated variants. PCAs that include PAHs and other heterocyclic compounds are also potentially harmful, but regulations generally focus on the overall "PCA extract" parameter as an indicator of carcinogenic contamination because a smaller amount of PCA extract ensures a correspondingly lower PAH content (European Parliament & Council, 2005). Due to the high toxicity profile of PAHs, regulations related to the content of PAHs in RPO products are more strictly enforced. If the PCA extract requirement (>3% mass) is not met but all PAH marker parameters are still within safe limits, extender oil can still be marketed provided that the manufacturer or importer can demonstrate compliance with the PAHs limit and retest

every six months or after major process changes (European Parliament & Council, 2005). This reflects that while PCAs provide indications of widespread aromatic contamination, regulators emphasize monitoring of PAHs specifically due to the high health risks they pose as a determining criterion for final compliance.

The manufacture of RPO products of the treated residual aromatic extract (TRAE) type can be a solution to overcome this. TRAE is an RPO product that results from the modification process of RAE products. The advantages of TRAE products are focused on their lower viscosity and good thermal stability. Good thermal stability is taken into account so that the product still has use value comparable to the RAE product. Low viscosity can improve the efficiency of the mixing and processing process, which in turn can also allow a reduction in the content of PAHs, thereby minimizing health risks as well as environmental impacts.

Several approaches have been investigated to reduce viscosity and aromatic content in heavy petroleum fractions. Alomair and Almusallam (2013) demonstrated that blending heavy crude oil with paraffinic base oils effectively reduces viscosity through dilution, achieving viscosity reductions of up to 70% depending on the mixing ratio and temperature conditions. Their study emphasized the importance of selecting compatible diluents to maintain mixture stability and prevent phase separation. Similarly, Mohammadi et al. (2019) investigated the rheological behavior of heavy crude oil diluted with lighter hydrocarbons, reporting that viscosity reduction follows logarithmic mixing rules under ideal conditions but exhibits deviation in systems with significant molecular interactions. These findings underscore the necessity of theoretical modeling approaches, such as the Grunberg-Nissan equation, to predict mixture viscosity accurately in non-ideal systems.

In the context of PAH reduction, Comber et al. (2013) evaluated various treatment methods for aromatic extracts used in rubber applications, noting that solvent extraction and hydroprocessing can reduce PAH content but often at the expense of aromatic functionality essential for rubber compatibility. Their work highlighted the challenge of balancing environmental compliance with performance requirements. More recently, Sanière et al. (2004) reviewed viscosity reduction techniques for crude oils, emphasizing that dilution with low-aromatic base stocks represents a cost-effective alternative to thermal or catalytic processing, particularly for applications where moderate aromatic content remains acceptable. However, limited research has systematically examined the use of environmentally compliant base lubricating oils as diluents specifically for RAE materials, particularly with comprehensive characterization of both rheological properties and PAH profiles across multiple formulation ratios. This gap is especially pronounced in studies that integrate theoretical viscosity prediction models with experimental validation and regulatory compliance assessment.

The novelty of this research lies in three key aspects: (1) the systematic application of the Grunberg-Nissan theoretical model to predict and validate the viscosity behavior of RAE-LBO binary systems across multiple formulation ratios, which has not been previously reported for TRAE formulations; (2) the use of HVI 60 base lubricating oil as

a diluent specifically selected for its low PAH content and chemical compatibility with RAE, representing an environmentally preferable alternative to conventional solvents; and (3) the comprehensive characterization framework that simultaneously evaluates critical parameters (viscosity, PCA, PAH) alongside supporting parameters (PNA composition, VGC, aniline point) to establish both regulatory compliance and functional performance benchmarks against commercial TRAE products.

The formulation of the traee product is developed through a viscosity modification process using the dilution method on RAE product materials. The dilution method allows the adjustment of the viscosity of TRAE by mixing RAE material with lube base oil. The selection of lube base oil as a dilution ingredient needs to be considered because it has the potential to reduce the content of PAHs due to its lower PAH content.

## **METHOD**

This study used blending equipment as the main tool, along with various supporting laboratory tools such as pipettes, beakers, analytical scales, and standard test equipment according to ASTM methods. The materials used included two types of residual aromatic extract (RAE) – A and B – and HVI 60 base lubricating oil as a solvent in the dilution method. The blending process was carried out at 100°C with a stirring speed of 200 rpm for 60 minutes. After blending, various physical tests such as density, viscosity, pour point, and flash point were performed using the appropriate ASTM methods. Additionally, the polycyclic aromatic (PCA) content was tested using DMSO and cyclohexane, while PAH and PNA contents were analyzed using GC-MS after a multi-stage purification process. This methodology optimized TRAE formulations while ensuring regulatory compliance and maintaining product safety and performance.

## **RESULTS AND DISCUSSION**

This internship activity is carried out with reference to the general and specific goals that have been set. The general purpose of the internship activity is to carry out parameter testing in accordance with the specifications of samples of Fuel Oil (BBM) products, namely Avtur and Gas Oil, as well as Non-Fuel products (NBBM), namely Rubber Process Oil. Through this activity, it is hoped to gain a comprehensive understanding of ASTM-standardized testing methods. The ASTM standards studied include scope, principles, and traceability aspects, especially in terms of reproducibility and repeatability. In addition, this activity also aims to increase participants' familiarity with the use of relevant laboratory instruments and foster awareness of the importance of the suitability of fuel and NBBM product specifications, both in terms of the testing process and comparison.

The specific purpose of the internship activity is to conduct research or research entitled TRAE Product Formulation Through the Dilution / Viscosity Modification Method of RAE Materials Using Basic Lubricating Oil This research is part of an internship activity that aims to formulate the formulation of Treated Residual Aromatic Extract (TRAE) products through the dilution method, namely the mixing of the main

ingredient Residual Aromatic Extract (RAE) with base lubricating oils in various ratios. TRAE is a product modified by RAE that has undergone special treatment, such as purification or mixing, to improve certain characteristics according to industrial needs. This research focuses on critical parameters, namely viscosity, percentage of Polycyclic Aromatic Compounds (%PCA), and Polycyclic Aromatic Hydrocarbons (PAHs) content, which are the main indicators of product quality. In addition, several supporting parameters were also analyzed to obtain a more comprehensive picture of the characteristics of the resulting formulation.

The research activity began with the process of identification and characterization of raw materials, followed by the formulation of RAE and basic lubricating oil. Each sample of the formulation is tested according to applicable standards, then compared with the TRAE product specifications of competitors as an evaluation reference. The ultimate goal of this study is to obtain a TRAE formulation that is able to meet or approach technical specifications in key parameters, and has potential applications according to industry needs.

### **TRAE product formulation through the Dilution / Viscosity Modification Method of RAE Material Using Base Lubricating Oil**

#### ***Test of Critical Parameters of Single Component RAE Materials and Base Lubricating Oil***

TRAE products are formulated with the formulation's main focus on viscosity control. Viscosity is one of the most crucial parameters in ensuring lubricant performance. Viscosity plays a crucial role in maintaining the stability of the lubricating film, minimizing friction, and ensuring optimal engine performance under various temperature and load conditions (Gao et al., 2023; Kobayashi et al., 2023). TRAE product formulation is aimed at producing low-viscosity lubricants whose viscosity values refer to the typical three commercial products, namely Orgkhim Norman 388, H&R Vivatec 700, and H&R Vivatec 800. The viscosity value of the final formula is expected to meet one of the specifications of the three reference products, or be within the range of viscosity values owned by all three. The target viscosity values based on the typical products of Orgkhim Norman 388, H&R Vivatec 700 and H&R Vivatec 800 can be observed in the table.

**Table 4. Kinematic viscosity of typical products**

Parameter	Units	Method	Orgkhim Norman 388	H&R Vivatec 700	H&R Vivatec 800
<b>Viscosite</b>					
<b>Kinematics at 40°C</b>	mm <sup>2</sup> /s	ASTM D-445	-	900	760
<b>Viscosite</b>					
<b>Kinematics at 100°C</b>	mm <sup>2</sup> /s	ASTM D-445	31,0	29,0	30,0

The main raw material in this formulation is RAE (Residual Aromatic Extract), which is the weight fraction of the by-product of the lubricant refining process, which is used in the manufacture of RAE products. This ingredient has high tackiness due to the high content of aromatic compounds. However, this RAE material has a relatively high viscosity, which can be an obstacle in its use as a flexible lubricant or in low-temperature

applications. Therefore, in order to modify the viscosity and make it conform to the specifications of modern lubricants, the right solvent material is required. In the test, two types of RAE materials with different production times were used. This is to determine that RAE materials have different parameter values at each production time.

In this case, lube base oil is used as the main solvent. His selection was based on several strategic considerations. The low viscosity of lube base oil allows the dilution process of the RAE material to reach the target viscosity effectively, without the need for further chemical reactions. Second, the PCA (Polycyclic Aromatics) content in lube base oil, which is usually much lower than RAE material, helps to lower the total PCA level in the final mixture to match the maximum limit allowed by environmental regulations. Third, the chemical compatibility between the base oil and similar RAE materials allows the two to mix homogeneously, creating a physically and chemically stable product.

The purpose of testing critical parameters on raw materials is to ensure that their characteristics are suitable for use as a base material in the formulation of TRAE products. The parameters tested are selected specifically based on the critical parameters targeted in the TRAE final product, namely viscosity and PCA content, to ensure technical performance and compliance with environmental regulations. Therefore, this test not only serves as a validation of the quality of raw materials, but also as the basis for the formulation of mixing strategies in the production process.

**Table 2. Single-Component Kinematic Viscosity Test Results of RAE Materials and Base Lubricating Oils**

Parameter	Units	Method	Test Results		
			Ingredients RAE A	Ingredients RAE B	Lube Base Oil
Viscosite Kinematics at 40°C	mm <sup>2</sup> /s	ASTM D-445	89,9	136,1	4,82
Viscosite Kinematics at 40°C	mm <sup>2</sup> /s	ASTM D-445	17987	42391	23,71
Polycyclic Aromatics	% wt	IP-346	13.67	14,1	1,15

Based on the test results presented in the Table, it can be seen that RAE material has a kinematic viscosity of 23.71 mm<sup>2</sup>/s at 40 °C and 8.62 mm<sup>2</sup>/s at 100 °C, and contains Polycyclic Aromatics (PCA) of 14.1%. On the other hand, lube base oil shows much lower viscosity characteristics, namely 4.82 mm<sup>2</sup>/s at 100 °C and 28.71 mm<sup>2</sup>/s at 40 °C, and has a much smaller PCA content of 1.15%.

PCA levels are measured to ensure that the resulting formulation does not contain excessive amounts of aromatic compounds, so that it still meets the sustainability and environmental aspects. In this case, lube base oil was chosen as a solvent because in addition to being chemically compatible with RAE components, it is also effective in lowering viscosity to a level that is in accordance with the final product specifications, while helping to lower PCA levels to meet environmental standards. With reference to the results of this table, the TRAE formulation is prepared to achieve the viscosity target, as well as ensure that the PCA content is in accordance with the provisions of the REACH

Regulation. This test aims not only to verify the quality of raw materials, but also to It is a crucial first step in determining the composition of the formulation that is balanced between technical performance and environmental safety.

The regulation of PCA levels measured by the IP-346 standard method using dimethyl sulfoxide (DMSO) extraction and refractive index measurements, must be below 3% of the product mass to be considered to meet safety standards. This PCA level is used as a practical indicator that represents the overall content of harmful aromatic compounds in petroleum products, making routine testing more efficient and effective. If PCA levels exceed the set limit, more specific testing of benzo[a]pyrene (BaP) content and total polycyclic aromatic hydrocarbons (PAHs) should be performed as a final verification step to ensure that the maximum limit of BaP does not exceed 1 mg/kg and that the total PAHs do not exceed 10 mg/kg are met. Thus, BaP and PAHs testing serves as an additional solution if the results of PCA measurements show nonconformity, maintaining product safety and compliance with regulations.

### **Analysis of the Relationship of Kinematic Viscosity with RAE:LBO Mixture Ratio Using Theoretical Models and Experimental Data as a Basis in TRAE Product Formulation**

The formulation of Treated Residual Aromatic Extract (TRAE) products developed from a mixture of RAE and lube base oil ingredients begins with testing of viscosity properties at various mixing ratios to evaluate the rheological behavior of the system. Tests were carried out on three main compositions with a mixing ratio of RAE ingredients: lube base oil, namely 85:15, 70:30, and 60:40. The selection of the ratio was based on an effort to evaluate the effect of increased RAE content on the physical and chemical properties of lubricants. The 85:15 ratio was chosen as a reference due to the still dominant lube base oil content, while the 70:30 and 60:40 ratios were used to see the extent to which increased RAE proportions could affect the overall performance of the lubricant.

Viscosity analysis is carried out using the Logarithmic Mixing Rule (Arrhenius-type) model approach or commonly known as the ideal logarithmic model, which is a model that assumes that the viscosity of the mixture is a logarithmic combination of the viscosity of each component without taking into account the interaction between molecules. This model provides a simple basic overview of the relationship between the viscosity of a mixture and its composition, especially when the components being mixed have similar characteristics and do not interact significantly.

$$\ln \eta_{mix} = x_1 \ln \eta_1 + x_2 \ln \eta_2 \quad (IV.1)$$

In the equation, the symbol  $\eta_{mix}$  represents the viscosity of the mixture of two components, while  $\eta_1$  and  $\eta_2$  are the viscosity of each pure component. The values of  $x_1$  and  $x_2$  indicate the mole fraction or volume fraction of the first and second components in the mixture, which must be equal to one. The symbol  $\ln$  is a natural logarithm, which is a logarithm based on the Euler number ( $e = 2.718$ ) which is used to represent the non-linear relationship of viscosity to the composition in a form that can be analyzed linearly

However, in complex blended formulations such as TRAE consisting of DAO and lube base oils with different chemical and physical characteristics, molecular interactions between components can significantly affect viscosity properties. Therefore, the Grunberg–Nissan model is used as an extension of the ideal model by adding the interaction parameter  $G$ . This parameter  $G$  represents the effects of intermolecular interactions that can cause the viscosity of the mixture to be higher or lower than the ideal model predicts.

$$\ln \eta_{mix} = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1 \cdot x_2 \cdot G \quad (IV.2)$$

By using the two models simultaneously, namely the ideal logarithmic model as the baseline and the Grunberg–Nissan model to account for molecular interactions, the viscosity analysis of the TRAE mixture becomes more comprehensive and accurate. This approach allows for a thorough evaluation of the rheological behavior of the mixture, so that the product formulation can be optimized to obtain the ideal viscosity properties to support maximum lubrication performance.

To support the analysis of the properties of the viscosity of the mixture, a plot was made that was compiled using the ideal logarithmic model. This plot shows the relationship between the mixed fraction of DAO : Lube Base Oil on the x-axis and the  $\ln$  value of the viscosity of the mixture on the y-axis. Based on the plot, an equation of lines  $y = ax + b$  is generated which is based on equation II.1 as can be observed below.

$$\ln \eta_{mix} = x_1 (\ln \eta_1 - \ln \eta_2) + \ln \eta_2 \quad (IV.3)$$

Furthermore, the actual viscosity value of the experimental results on the same composition is plotted on the graph as a data point. To account for deviations from the ideal model due to molecular interactions, the Grunberg–Nissan model is used. The  $G$  parameter is calculated based on the difference between the experimental value and the ideal model result in each composition. The  $G$ -value obtained varies for each composition, so it cannot be represented as a single constant value in the entire range of compositions.

Viscosity properties testing was performed on RAE A and Lube Base Oil (LBO) material mixture products with mixed volume variations of 85:15, 70:30, and 60:40. The results of this viscosity test were used to see the trend of viscosity changes in the composition, which was then used as a reference in formulating a mixture of RAE B with the same LBO. Since the type of LBO used is fixed, the characteristics of the solvent in the mixing system do not change. If the physicochemical characteristics of RAE B are not is far different from RAE A, so the viscosity trends resulting from the RAE A:LBO mixture can be used as an initial approach to predict the viscosity behavior of the RAE B:LBO mixture, before further testing is carried out.

The table of kinematic viscosity test results of the RAE A and LBO blending variations can be observed in the following table.

**Table 3. Kinematic viscosity test results of RAE A and LBO formula variations**

Parameter	Units	Method	Racing				
			Bahan RAE A : Lube Base Oil				
			100:0	85:15		70:30	60:40
			Material RAE A Murni	Lube Base Oil Murni			
Kinematic Viscosity 40°C	mm <sup>2</sup> /s	ASTM D-445	17987	1445,42	757,1	353,3	23,71
Kinematic Viscosity 100°C	mm <sup>2</sup> /s	ASTM D-445	89,9	46,67	23,88	17,60	4,82

Kinematic viscosity parameters are measured at temperatures of 40 °C and 100 °C. The measurement and specification of kinematic viscosity is determined at this temperature because at this temperature the lubricant undergoes a significant change in flow properties, and becomes a reference for international standards for comparing fluid characteristics. A temperature of 40 °C reflects normal operating conditions, while 100 °C reflects more extreme working conditions of machinery or equipment.

The data obtained in the table can be used in the creation of plot graphs based on the ideal logarithmic model. So that two graphs can be made that explain the character of the natural logarithmic relationship of kinematic viscosity at temperatures of 100 °C and 40 °C to fractions a mixture of RAE A and Lube Base Oil. This aims to determine the pattern of viscosity changes to variations in blending composition, which is very important in the formulation process.

The following is a graph of the relationship between kinematic viscosity at a temperature of 40 C and the faction fraction of the RAE:LBO material.

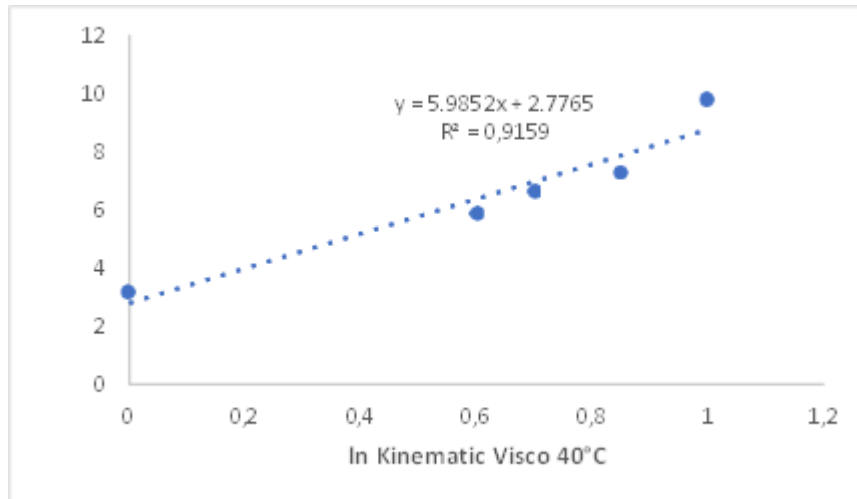


Figure 1. Graph of the relationship between kinematic viscosity 40°C vs Ratio of RAE material formula A:base lubricating oil

The graph shows the relationship between the value of kinematic viscosity at 40 °C and the mixed fraction of the RAE:LBO material. The results of linear regression on the experimental data produced the equation  $y=5.9852x+2.7765$  with a determination coefficient of  $R^2=0.9159$ . Although the  $R^2$  value is relatively high and shows a strong correlation, some data points experience deviations from the regression line. This

indicates that linear relationships do not fully accurately describe the behavior of the system. This deviation is thought to occur due to the existence of non-ideal interactions between the RAE and LBO molecules, which are not accounted for in a simple linear regression model. Based on the Grunberg–Nissan theory, the viscosity of the mixture is determined not only by the logarithmic contribution of each component, but also by G interaction parameters that reflect the attraction forces between different molecules (Grunberg & Nissan, 1949). The varying G-values for each composition indicate that the interactions between molecules are complex and non-uniform.

The regression result line equation allows an estimate of the mixed fraction of the RAE:LBO required to achieve a specific viscosity target. By converting the target viscosity value into a natural logarithm (ln) form, the mixed fraction can be calculated directly using the ideal linear regression equation. Based on this approach, the fraction of the RAE material is estimated to achieve kinematic viscosity at a temperature of 40 °C which is close to the typical value of some reference products. The viscosity target of 900 mm<sup>2</sup>/s was chosen as a representation of the H&R Vivatec 700 product, while the value of 760 mm<sup>2</sup>/s represents the H&R Vivatec 800. For the Orgkhim Norman 388 product, there is no kinematic viscosity target specification at 40 °C that can be used as a reference. From the calculation results, it was obtained that the mole fraction of RAE material needed to achieve a viscosity of 900 mm<sup>2</sup>/s is about 0.67, while for a viscosity of 760 mm<sup>2</sup>/s it is about 0.64. Thus, the mixed formulation can be compiled using the RAE:LBO ratio of 67:33 and 64:36 respectively. Furthermore, in order to consider the optimal formulation, it is necessary to observe the graph of the relationship between the target kinematic viscosity at a temperature of 100

**The graph of the relationship can be observed in the following figure.**

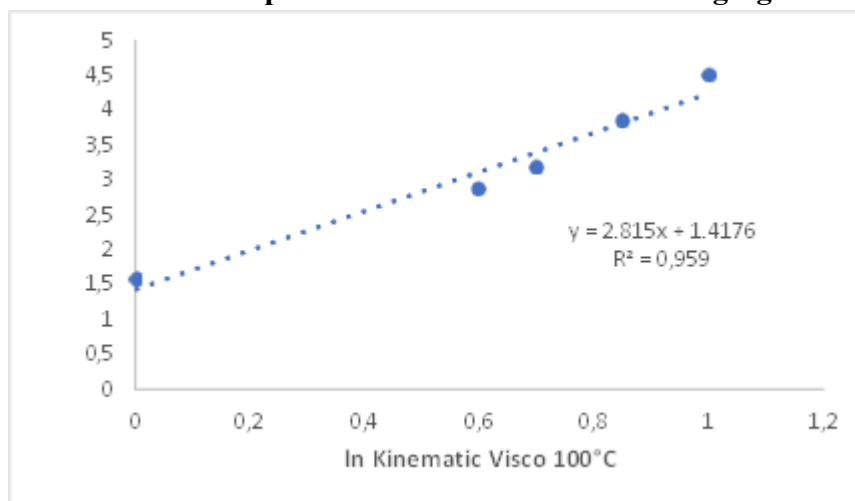


Figure 2. Graph of the Relationship of 100°C Kinematic Viscosity Vs Formula Ratio of RAE Material A:Base Lubricating Oil

The graph shows the relationship between the value of kinematic viscosity at 100 °C and the mixed fraction of the RAE:LBO material. The results of linear regression on the experimental data produced the equation  $y=2.815x+1.4176$  with a determination

coefficient of  $R^2=0.959$ . Although the  $R^2$  value shows a high fit, there are still minor deviations from the ideal model. Just like the previous graph (at 40 °C), this deviation is thought to be due to the presence of non-ideal interactions between molecules in the mixture. However, compared to lower temperatures, the effect of this interaction appears to decrease at 100 °C. This indicates that the increase in temperature can improve the mixing uniformity of the two components, thereby reducing the deviation from the linear model.

Using the same calculation method, the fraction of RAE material required to achieve the kinematic viscosity target at 100 °C can be estimated. The viscosity targets used reflect the typical values of some of the reference products, which are 31.0 mm<sup>2</sup>/s for the Orgkhim Norman 388, 29.0 mm<sup>2</sup>/s for the H&R Vivatec 700, and 30.0 mm<sup>2</sup>/s for the H&R Vivatec 800. Based on the calculation of the regression equation, it is obtained that the mole fraction of the RAE material needed to achieve the viscosity of 31.0 mm<sup>2</sup>/s is about 0.72; for 29.0 mm<sup>2</sup>/s about 0.70; and for 30.0 mm<sup>2</sup>/s about 0.69. Thus, the mixed formulation can be arranged with a RAE:LBO ratio of 72:28, 70:30, and 69:31 respectively.

Based on the calculations from the two graphs (at temperatures of 40 °C and 100 °C), five RAE:LBO material formulation ratios that can be used to approach the viscosity of the reference product are obtained, namely 64:36, 67:33, 69:31, 70:30, and 72:28. Nevertheless, this whole calculation is still based on the assumption of the ideal mixture, without taking into account the molecular interactions between the components. In order to obtain a more precise and realistic formulation, it is necessary to correct the deviations that may occur due to such non-ideal interactions, for example through the application of the Grunberg–Nissan model (Grunberg & Nissan, 1949), which specifically takes into account the parameters of interaction between two types of molecules in the viscosity mixture.

### **Selection of Formulation Composition Variations and Complete Parameter Test Plan**

The formulation was carried out using RAE B material, which characteristically has a higher viscosity than RAE A used in the initial stage of predictive analysis. Therefore, although modeling of the viscosity-to-mixture ratio relationship was done based on the mixing data of RAE A with LBO, the results can still be used as a preliminary approach. Assuming that the viscosity trend follows a similar pattern, three main variations of the composition of RAE B:LBO, namely 75:25, 72:28, and 70:30, were selected to be formulated and further tested through complete parameter testing. The 72:28 and 70:30 compositions were chosen because the results of previous regression calculations showed the closest compatibility with the viscosity of reference products such as Orgkhim Norman 388, H&R Vivatec 700 and Vivatec 800 at 100 °C. Meanwhile, the 75:25 composition is used as an exploratory formulation with higher RAE B content, to observe the upper limit of viscosity while evaluating the stability and suitability of the mixture's performance over a wider range of characteristics.

To ensure that the developed TRAE formulation products meet the expected technical performance and chemical characteristics, a series of tests were carried out against 11 key parameters. The purpose of this test is to evaluate the physicochemical characteristics of each TRAE formula produced, as well as to compare it with commercial reference products that have been used previously as a reference in determining product viscosity, namely Norman 388, Vivatec 700, and Vivatec 800.

In the process of formulating TRAE with the dilution method on RAE materials using LBO, the selection of test parameters is carried out thoroughly to describe the performance, safety, and aromatic characteristics of the product. Each parameter was chosen because it made an important contribution to the technical functionality and commercial feasibility of the product. The critical parameters that are the focus of the evaluation are kinematic viscosity at temperatures of 40 °C and 100 °C, as these two parameters are the main targets in the formulation of TRAE to ensure the suitability of lubricant performance with the expected standard. In addition to viscosity, other critical parameters are the percentage of Polycyclic Aromatics (%PCA) as well as the content of Polycyclic Aromatic Hydrocarbons (PAH), specifically Benzo[a]pyrene (BaP) compounds and total

$\Sigma$ 8PAH. The assessment of these parameters aims to ensure compliance with international regulations, in particular the EU's REACH Regulation (EC No. 1907/2006) which limits the maximum PCA content to 3% w/w based on the IP 346 method, as well as the EU Directive 2005/69/EC, which regulates the restriction of 8 types of PAH (including BaP) in materials such as solvent oils and extender oils used in rubber or tire products, with a certain maximum limit for each compound. In practice, there is a connection between PCA and PAH parameters. Based on the regulations in EFSA (2008) and Commission Regulation (EU) No. 835/2011, a product that does not meet the PCA threshold can still be considered for production if the value of PAH, especially BaP and  $\Sigma$ PAH, is in accordance with the provisions of applicable international regulations.

Other parameters in this analysis are supportive and play a role in supporting performance evaluation and product safety aspects. The Pour Point parameter indicates the lowest temperature when the oil can still flow, while the Flash Point indicates the minimum temperature the oil vapor can burn, both of which are important for applications at low temperatures and storage safety (ASTM, 2020). Aniline Point and Viscosity Gravity Constant (VGC) describe the level of aromaticity and hydrocarbon structure in the product (Speight, 2014; Chilingarian et al., 1987). The Refractive Index reflects the density of electrons and is closely related to the aromatic properties of the oil. The %PNA (paraffinic, naphthenic, aromatic) parameter indicates the composition of the type of hydrocarbons that affect viscosity, solvent properties, and toxicity (CONCAWE, 2019). In addition, specific gravity, which basically only gives an idea of the relative density of oil to water, is also included as complementary data in the calculation of VGC and %PNA (Speight, 2014). Although they are only supportive, all these parameters make an important contribution to understanding the overall characteristics and final performance of the product that has been formulated.

**Discussion of Complete Parameter Test Results in TRAE Formulation**  
**Critical Parameter Test Results**

**Table 4. Critical Parameter Test Results**

Typical Comparator			Test Results					
Parameter	Units	Method	Norman 388	Vivatec 700	Vivatec 800	70:30	72:28	75:25
% Polycyclic Aromatics	%wt	IP- 346	To be reported	To be reported	To be reported	6,8	11,45	<b>11,6</b>
Viscosity, Kinematic at 100 °C	mm <sup>2</sup> / s	ASTM D-445	31,0	29,0	30,0	31,41	34,18	<b>38,32</b>
Viscosity, Kinematic at 40 °C	mm <sup>2</sup> / s	ASTM D-445	-	900,0	760,0	799,0	1166,0	<b>1416,0</b>
BaP	ppm wt		<1.0	<1.0	<1.0	0,13	0,14	<b>0,17</b>
PAH								
Σ8PAH	ppm wt	MS- Yes	<10	<10	<10	1,57	1,85	<b>1,94</b>

The kinematic viscosity parameter is the main parameter that is used as a target in formulating TRAE products by the viscosity modification method, namely through dilution techniques. Kinematic viscosity itself is one of the important indicators in assessing the performance of oil or base oil, especially related to flow stability and lubrication ability at various operating temperatures. Based on the test results, kinematic viscosity values at 100 °C for 70:30, 72:28, and 75:25 formulations

They are 31.41 mm<sup>2</sup>/s, 34.18 mm<sup>2</sup>/s, and 38.32 mm<sup>2</sup>/s, respectively. When compared to typical data of comparison products such as Norman 388 (31.0 mm<sup>2</sup>/s), Vivatec 700 (29.0 mm<sup>2</sup>/s), and Vivatec 800 (30.0 mm<sup>2</sup>/s), then the 70:30 formulation indicates a value that closest to typical viscosity characteristics, particularly to Norman 388. Meanwhile, the 72:28 and 75:25 formulations show significantly increased viscosity values, which may indicate over-viscosity and potential decreased flow efficiency in certain applications that require fluids with lighter flow characteristics.

At 40 °C, a similar trend is also seen. The 70:30 formulation has a viscosity of 799.0 mm<sup>2</sup>/s, identical to Vivatec 700 and quite close to the target range of similar products. The 72:28 (1166.0 mm<sup>2</sup>/s) and 75:25 (1416.0 mm<sup>2</sup>/s) formulations show a sharp increase, which may be less than ideal if product specifications require fluid characteristics to be close to industry standard viscosity values. Thus, from the point of view of kinematic viscosity at both high and low temperatures, the 70:30 formulation exhibits the closest

performance to the typical comparator product and can be considered the most balanced formula candidate in terms of flow and viscosity.

In addition to viscosity parameters, the content of Polycyclic Aromatics (PCA) is a critical aspect in the formulation of TRAE products, especially related to environmental and occupational health safety issues. Based on the test results, the PCA content in the 70:30, 72:28, and 75:25 formulations was 6.8%, 11.54%, and 11.60% by weight, respectively. An increase in PCA content as the proportion of TRAE increases is natural, considering that TRAE naturally contains high aromatic fractions. However, although the difference is relatively small (less than 0.4%), the overall values still go far beyond the limits set in a number of international regulations.

According to Annex XVII of REACH (Regulation (EC) No 1907/2006), mineral oil-based materials that have a PCA content of  $\geq 3\%$  by weight (IP 346 method) are classified as Carcinogenic Category 1B, so its use is restricted to non-consumer applications and requires appropriate hazard marking. Thus, the three formulations of TRAE tested fall under the CMR (Carcinogenic, Mutagenic, and Reprotoxic) category and cannot be used freely without additional evaluation.

For this reason, it is recommended to carry out further testing to find out more specifically the profile of individual aromatic compounds in the PCA fraction, especially the Polycyclic Aromatic Hydrocarbons (PAH) group which has a higher level of toxicity. Additional testing may include analysis of BaP (Benzo[a]pyrene) and PAH  $\Sigma 8$  according to the GC-MS method as well as toxicology tests if the product is intended for more sensitive applications. This more in-depth evaluation is important not only to meet safety standards, but also to ensure compliance with regulations in destination markets, such as REACH, OSHA (US), and potential Minister of Environment and Forestry (MoEF) regulations in Indonesia.

As a continuation of the PCA analysis, testing of Polycyclic Aromatic Hydrocarbons (PAH) compounds was conducted to assess the specific toxicity potential of heavy aromatic fractions in formulations. The parameters tested included Benzo[a]pyrene (BaP) as a key indicator of carcinogenic compounds, as well as  $\Sigma$ PAH8RES, which is the number of the eight PAH compounds considered the most relevant according to the European Chemicals Agency (ECHA).

The test results showed that the BaP content in the 70:30, 72:28, and 75:25 formulations was 0.13 ppm, 0.14 ppm, and 0.17 ppm, respectively. All of these values are well below the maximum threshold of 1 ppm (1 mg/kg) set out in REACH Annex XVII, Entry 50, which states that the product should not be marketed for use in extender oil in the manufacture of tires or parts of tires if it contains more than from 1 mg/kg BaP or more than 10 mg/kg of the total amount of PAH registered.

For the  $\Sigma 8$ PAH parameter, the results obtained were 1.54 ppm (70:30), 1.85 ppm (72:28), and 1.94 ppm (75:25). All of these values are still well below the 10 ppm threshold set for non-consumer materials, such as industrial lubricants, process oils, and rubber raw materials. Thus, all three formulations meet safety standards and comply with EU environmental and health regulations. Overall, however, the 70:30 formulation still

showed excellence in terms of performance and environmental acceptability, with the lowest BaP content and total PAH among the three formulations tested.

### Supporting Parameter Test Results

Table 5. Supporting parameter test results

Parameter	Units	Method	Typical Comparator			Test Results		
			Norman 388	Vivatec 700	Vivatec 800	70:30	72:28	75:25
Relative Density at 15.6/15.6 °F	-	ASTM D-1298	-	-	-	0,9639	0,9674	0,9712
Bias Index at 20 °C	-	ASTM D-1218	-	1,526	1,536	1,544	1,546	1,549
Viscosity Gravity Constant	-	ASTM D-2501	0,883	0,884	0,897	0,902	0,905	0,909
Titik Aniline	°C	ASTM D-611	75,0	81,0	75,0	70,9	69,4	67,6
%P	%		-	43,0	42,0	42,1	40,0	43,9
PNA %N	%		-	36,0	29,0	24,0	26,8	22,3
%A	%		26,0	21,0	29,0	33,9	33,2	33,8
Titik Tuang	°C	ASTM D-97	-	3	6	12	12	9
Titik Nyala	°C	ASTM D-92	-	240	260	226	233	239

As a complement to the critical parameter analysis, testing of a number of supporting parameters is carried out to assess the stability and functional characteristics of each formulation. Thermal performance evaluation is carried out through pour point and flash point analysis, which are the main indicators of formulation stability at extreme temperatures. The 70:30 formulation as the selected formula has a pour point value of 12°C, the same as the 72:28 formulation, while the 75:25 formulation is lower at 9°C. The decrease in the pour point along with the increase in the ratio of the base lubricating oil is not significant, considering that 70:30 and 72:28 have identical values. Compared to the typical Vivatec 700 and 800 which have pour points of 3°C and 6°C respectively, the 70:30 formula shows limited flow performance at 12°C and is less than optimal at lower temperatures.

In the flash point aspect, the 70:30 formula has a value of 226°C, which is lower than the 72:28 (233°C) and 75:25 (239°C) formulations. This decrease in flash point indicates that an increase in the ratio of light lubricating oils increases product volatility

so that the flash point decreases. When compared to the typical Vivatec 700 and 800 which have flash points of 240°C and 260°C, the 70:30 formula shows lower thermal stability. Thus, the thermal performance of the selected 70:30 formula is limited to a maximum temperature of 226°C.

The aromaticity characteristics of each formula can be evaluated through the parameters of Aniline Point, Viscosity Gravity Constant (VGC), Refractive Index, Specific Gravity, and PNA (Paraffinic, Naphthenic, Aromatic) composition. The results showed that all three formulas had a higher level of aromaticity compared to the typical comparator. This is demonstrated by lower Aniline Point values, which are 70.9 °C in the 70:30 formula, 69.4 in the 72:28 formula and 67.6 °C in the 75:25 formula. The aniline point value indicates the dissolution interaction between the oil and aniline, so the lower the aniline point value, the more aromatic content in the product. Based on this statement, the data on each formula shows a decrease in the aromaticity of the product along with the increase in the ratio of base lubricating oils in the product formula. When compared to typical ones such as Norman 388 (75.0 °C), Vivatec 700 (80.0 °C), and Vivatec 800 (75.0 °C), it can be concluded that the aromaticity levels of all three formulas are higher compared to the typical ones.

This is also supported by other parameters such as the Viscosity Gravity Constant (VGC), which shows a tendency to increase in value as the DAO ratio in the formula increases, from 0.905 in the 70:30 formula to 0.909 in the 75:25 formula. A high VGC value is an indicator of the dominance of aromatic ring structures in the composition of hydrocarbons. In comparison, typical VGC values such as Norman 388 (0.883), Vivatec 700 (0.884), and Vivatec 800 (0.897) are still lower than the three formulas, reinforcing the conclusion that blended products have a higher aromatic content than commonly used commercial products.

This trend is also in line with the Refractive Index which increased from 1.5440 in the 70:30 formula, 1.5260 in the 72:28 formula, and 1.5490 in the 75:25 formula. This value is greater than typical refractive indices such as Vivatec 700 (1.526) and Vivatec 800 (1.536). The refractive index value indicates the electron density and polarity of the molecules inside the product. Higher refractive index values indicate higher electron density and polarity, which are characteristic of aromatic compounds.

Furthermore, the Specific Gravity (SG) value also shows a parallel trend, which is an increase from 0.9639 in the formula 70:30, 0.9674 on the formula 72:28, and 0.9712 . Higher specific gravity is an indicator of the density of the molecular structure, which is generally positively correlated with a high content of aromatic and naphthenic compounds. The SG value of this blended formula is consistently higher, indicating the presence of more complex and heavy hydrocarbon structures, especially from the aromatic group. However, the SG value has no reference to a typical product because it is basically just complementary data to determine %PNA and VGC Parameters.

Furthermore, the evaluation is also based on the composition of PNA (Paraffinic, Naphthenic, Aromatic), the data show that the aromatic fraction does not experience a trend in changes in ratio, value The %aromatic is 33.9% in the 70:30 formula to 33.2%

in the Formula 72:28, and 33,8 in Formula 75:25. However, if Compared to the typical Norman 388 (26.1), Vicatek 700 (21.0), Vivatec 800 (29.0), the aromatic value of each formula is still higher and the data is in line with other parameters.

In terms of performance, the high aromatic content provides advantages in terms of solubility to resins, polymers, and natural or synthetic rubber, which are needed in product formulations such as extender oils for the tire industry and other rubber products. In addition, the softening properties and mechanical flexibility enhanced by the aromatic fraction contribute positively to the quality of the final product. However, this performance advantage needs to be balanced with consideration of environmental and health aspects, especially if the product contains significant amounts of polycyclic aromatic compounds (PAHs). As discussed in the previous test.

Taking overall performance into account, the 70:30 formula shows a good balance of functionality and chemical characteristics when compared to typical products such as Norman 388, Vivatec 700, and Vivatec 800. Although the pour point (12 °C) and flash point (226 °C) values are still below Vivatec's superior thermal standards (3–6 °C and 240–260 °C), the 70:30 formula remains adequate for applications at medium temperatures. Its main advantage lies in its higher aromatic content—demonstrated by a lower Aniline Point value (70.9 °C), a higher VGC (0.905), a refractive index and specific gravity that exceeds that of a typical product, and an aromatic fraction of 33.9% that is consistently higher than typical (21.0–29.0%). This supports its performance as an effective oil extender, especially in the dissolution of resins, polymers, and elastomers. However, the high content of aromatic compounds also requires attention to environmental and health aspects, especially the risk of the presence of potentially toxic polycyclic aromatic compounds (PAHs). Therefore, although the 70:30 formula excels in functionality compared to typical products, its use must be accompanied by strict quality control. Based on the results of previous tests, the PAH content in this formula has met the applicable specifications, so the product is declared safe for industrial applications according to the set environmental standards.

## **CONCLUSION**

Analysis of residual aromatic extract (RAE) characteristics revealed high viscosity and PCA content, making it suitable for rubber processing oil (RPO) after modification. Dilution with base lubricating oil successfully produced treated residual aromatic extract (TRAЕ) with targeted viscosity values, effectively reducing PCA and PAH content to improve environmental and occupational safety. The optimal 70:30 RAE-to-base oil ratio met critical specifications across key parameters while balancing essential aromatic properties for rubber industry performance. Future research should investigate long-term stability, rubber compound compatibility testing, and scale-up production trials to validate industrial applicability.

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