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RESEARCH PAPER

Study of inorganic based anti-blocks as migration control of slip additive on surface polyethylene monolayer film

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Abstract: Slip migration must be controlled to maintain the performance and quality of flexible packaging. Inorganic based anti-block materials can be used to control the slip migration. This paper reported the effect of anti-block type on the inhibition of slip migration on polyethylene monolayer film. A series of formulations were made with three different anti-block additives, namely talc, natural silica, and synthetic silica, along with erucamide. The optical properties (haze) and friction properties (COF) were measured to assess the film characteristics as the development of slip migration in the presence of anti-block additives. Characterization of the anti-block material was conducted by SEM-EDX, slip additive type was examined by GC-MS, while the slip content on the surface was analysed by FTIR. The result showed that after seven days, synthetic silica anti-block gives COF up to 0.095, with the trace erucamide content on the film surface of 394 ppm, the lowest amongst other types of the anti-block used. The smaller particle size and higher silica content on synthetic silica anti-block resulted in better friction properties which act as a good barrier to limit a migration of erucamide onto the film surface.

Keywords: inorganic anti-block, erucamide, migration, coefficient of friction, haze.

INTRODUCTION

Plastic packaging is an essential material in the food industry to protect the food from external factors (such as humidity, temperature, odours, shock, physical damage, microorganisms, and dust) and to maintain its physical, chemical, and microbiological properties; as a result, it can extend their shelf life [1]. Plastic packaging is used widely in the food industry due to its considerable properties, such as inexpensive, lightweight, flexible design, heat sealable, easy to print, and it can be integrated with another process where the package is formed, filled, and sealed in the same production line [2]. Plastics are made from polymers that commonly incorporate additives to enhance the processability, performance, properties, characteristics, or appearance according to the end use. Additives are a vital component in polymer manufacture, which act as processing aids during shaping or production and ensure the long-term performance of the final product. Some additives include antioxidants, antifogging agents, emulsifiers, lubricants, anti-blocking agents, slip agents, plasticizers, release agents, solvents, stabilizers, thickeners, and UV absorbers. Additives are commonly used in the form of masterbatches, which contain a high dosage of additives that have already been dispersed in the parent polymer. Masterbatch has a lot of advantages over directly-used additive, such as good ability of dosing, ease of use, homogeneous mixing, and less dust (concerning safety) [3].

Plastic packaging systems are ubiquitous in manufacturing and require some technical needs that must be met by the plastic packaging itself. Adequate level of machinability of the film on high-speed packaging lines is an essential requirement in plastic packaging manufacture. In flexible packaging

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manufacture, the film must glide easily over film and metal surfaces. The slipperiness or tackiness of a packaging film affects its ability to be handled on high-speed packaging lines. To reduce the handling problems on the high-speed packaging lines, plastic film requires a minimum level of friction. There are two related but independent properties that are important in flexible packaging manufacture, namely: coefficient of friction (COF) and anti-blocking behaviour. The COF is the ratio of the force required to slide a film over a surface to the total force perpendicular to that surface. It is related to how easily the film slides over other films or metal surfaces. The anti-blocking behaviour relates to how easy the film roll is released or unwind. The polymers with high COF usually tend to block, but this is not always the case. The types of additives used to control COF are usually not the same as those used to prevent blocking, and the two may be synergistic or antagonistic [4].

The widespread practice in the flexible packaging industry is to control and reduce friction coefficient by adding fatty acid amides called slip additives. Slip additives will form a microcrystalline structure or a lubricating layer on the film surface that effectively separates adjacent film layers and decreases film friction. Primary fatty acid amides such as Erucamide and oleamide are the most used slip additives. Erucamide is an amide derived from C22 monounsaturated erucic acid, while oleamide is derived from C18 monounsaturated oleic acid [5].

Slip is commonly added in the molten stage, and due to the insufficient compatibility with the matrix polymer, the amide of slip additive migrates to the film surface (solid stage)3. These slip agents on the packaging films are not always easy to control because of various factors affecting the additive migration. The unpredictability of slip additive migration, which is frequently influenced by environmental and formulation variables, can result in uncertain friction properties of the film, and impact the downstream operations and loss of productivity on the packaging lines generally go through, such as sealing, printing, and handling [6]. One of the practical ways to control the slip additive migration is by combining an inorganic material (such as an anti-block) and fatty acid amide-type slip agent to provide the targeted COF at lower loadings and give lower slip additive on the film surface. The different types of anti-block used, such as talc mineral, silica mineral, and synthetic silica, affected the film quality and properties. A few studies on the additive-additive interaction of erucamide and anti-blocking agents have shown that there is antagonism and synergism between erucamide and the type of inorganic anti-block that results in the development of COF and optical properties of film packaging. Radosta and Riley reported a reduction in COF of LDPE film using talc anti-block several days after film fabrication [7]. Many attempts have been done by the researcher to study

the migration of slip on the surface. Dulal et al. studied the migration of erucamide and behenamide on HDPE closure by FTIR and AFM [8]. Ningrum et al. evaluated the migration behaviour of erucamide and stearyl erucamide on PE film [9]. John et al. characterized an anti-block film containing erucamide but only examined the result properties [10]. Janorkar & Douglas examined COF film containing erucamide and silica loading on multilayer LDPE, but the migration of slip was not discussed yet [11]. However, the reason and discussion of how anti-block additives can control the migration of slip additives has yet to be widely reported.

In this paper, study of migration of the erucamide slip additive in the presence of an inorganic anti-block additive on monolayer polyethylene film is reported. Inorganic anti-block additives with different types and characteristics were presented as the factors that influence migration slip on film surfaces.

MATERIALS AND METHODS

Material and Instrumentations

Slip additive of a mixture between Erucamide and a variant of anti-block type additives (talc, silica mineral, and synthetic silica) was used in the form of a polyethylene-based masterbatch supplied by PT. LyondellBasell (Indonesia). Let-down resin Low-Density Polyethylene (LDPE) (0.918 g/cm^3) was used as a carrier resin for filmmaking with a melt flow rate (190°C, 2.16 kg) of 3.5 g/10 min obtained from The Polyolefin Company (Singapore).

The anti-block masterbatch was heated in the oven at 600°C for 5 min to characterize the morphology of anti-block additives by SEM (FEI Quanta 250 - Oxford) and by xT microscope control software. The element content in each anti-block was examined by EDX (Penta FET x-act Oxford Instrument) using AZtecOne software. The FTIR (Perkin Elmer) equipped with ATR was used to characterize the functional groups of the anti-block additives (650-4000 cm^{-1}).

Film Preparation

All additive masterbatches were blended to LDPE carrier resin to achieve the final loading of 1500 ppm of erucamide and 2000 ppm of variant anti-block additives (**Table 1**). Each film formulation was prepared using a single-layer cast film coextrusion line (Collin LCR-300 series) and set for 50 µm final thickness. The machine condition of cast film was shown in **Table 2**. LDPE resin containing only slip additive was extruded as a reference to study the impact of slip additive without anti-block additive

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(Formula A). Each film was stored in a humidity chamber at 23 °C RH 50% for further testing.

Table 1. Film formulation

Formulation	Slip additive		Anti-block additive	
	Type	Dosage in film (ppm)	Type	Dosage in film (ppm)
Film A	Erucamide	1500	-	-
Film B	Erucamide	1500	Talc	2000
Film C	Erucamide	1500	Silica mineral	2000
Film D	Erucamide	1500	Synthetic silica	2000

Table 2. Machine Condition

Parameter	Value
Zone 1-5 barrel temperature	(190 – 190 – 190 – 190 – 190) °C
Speed screw	30 rpm
Screw torque	63
Cooling water system	28 °C
Master roll	2.8 m/min
2nd roll	2.9 m/min
3rd roll	7%

Measurement of slip additive content on film surface

A 2.5 g film sample was cut into small pieces and then mixed and stirred with 80 ml of dichloromethane in 250 mL Erlenmeyer for five minutes. The solutions were then filtered off and evaporated using TurboVap (Caliper LifeSciences) with nitrogen gas flow 9-12 bar in the water bath temperature at 60 °C until the volume decreased to 5 mL. The solution was analyzed using GC-MS to confirm the erucamide slip additive type and FTIR to measure the erucamide content. The type of slip additive was examined by GC-MS (Agilent technologies 5975C) with capillary model column HP-1 MS (19091S-833) using chemstation software. The slip additive analysis by GC-MS was carried out at 50 °C initial oven temperature (hold for 1 min) and ramped 10 °C/min to 300 °C and held for 30 min. A 100 µL of pyrene was added to the solution sample as an internal standard for GC-MS analysis. For slip additive content, the IR spectra were recorded on Perkin Elmer's Spectrum One in a cuvette. Spectrum Quant software was used for data analysis. The amide absorption peak around 3200-3400 cm^{-1} (in Erucamide) was used for characterization. The amount of erucamide in the sample is reported as a concentration ratio, which is the absorbance of the sample divided by the absorbance of the erucamide solution standard at a peak of 3407 cm^{-1}. The concentration of erucamide in the sample was calculated using Eq. (1):

$$C\,sam = \frac{A\,sam}{A\,std} \times C\,std \qquad (1)$$

note: C_{sam} is the sample concentration (ppm), C_{std} is the erucamide standard concentration, A_{sam} is the absorbance of the sample, and A_{std} is the absorbance of erucamide standard at peak of 3407 cm^{-1}.

Film Characterization

Film characterization includes optical properties and COF values. The optical properties of the film were measured using a haze meter (BYK haze-gard instrument) based on ASTM D-1003-00. The COF values were determined using Thwing-Albert Instrument FP-2260 based on ASTM D1894. The test is carried out at room temperature, with a test speed of 150 mm/min, static test distance of 15 mm, and kinetic test distance of 115 mm. The COF measurement used a 200-gram mass sled and a 2000-gram load cell. MAP-COF software was used for data analysis.

RESULTS AND DISCUSSION

Characterization of Anti-block Additives

The anti-block additives are commonly used for PE in a certain amount because the PE film tends to stick to each other. The different anti-blocks used in PE will give different end anti-block properties of PE film. Talc is categorized as a clay-based hydrated magnesium silicate and belongs to the phyllosilicate group with the chemical formula of $Mg_3Si_4O_{10}(OH)_2$. Talc consisted of a layered structure like a mica [12]. Synthetic silica has a chemical formula of SiO_2 and an amorphous form, while natural silica is a sedimentary rock that consists of a single cell of diatoms [13].

Table 3. Element compositions analysis, statistical mean and standard deviation of anti-block particle size.

Anti-block additives	Mean particle size (µm)	STD deviation	Element composition (wt%)			
			Si (%)	O (%)	C (%)	Mg (%)
Talc	2.58	0.66	10.34	46.76	34.93	7.97
Silica mineral	8.95	3.21	20.66	44.65	34.69	-
Synthetic Silica	4.89	0.61	29.66	55.85	14.49	-

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SEM images were taken to characterize the size, structure, and morphology of anti-block additives used in this experiment. As shown in **Figure 1**(a), talc has a thin sheet-like flake plane with particle size from 2.4 to 3.4 µm, which was the lowest size than other used anti-block additives. This layer of talc is bonded in weak Van der Waal's forces and easy to slide over each other, giving characteristics of softness [14]. Natural silica type quartz has a different morphology from synthetic silica, in which synthetic silica has a smooth smooth surface, whereas natural silica has coarser and rough surfaces. The natural silica also has a random particle distribution size from 2.0 to 18.0 µm. In contrast, synthetic silica has a fine distribution particle size of 4.4-5.5 µm. From the SEM-EDX result, synthetic silica has the highest silica content, followed by natural silica and talc. In the element content of talc, there is magnesium, which is not found in natural and synthetic silica due to the kind of clay mineral anti-block type of talc. This characterization of this anti-block material will support the analysis of the effect of slip migration in the discussion.
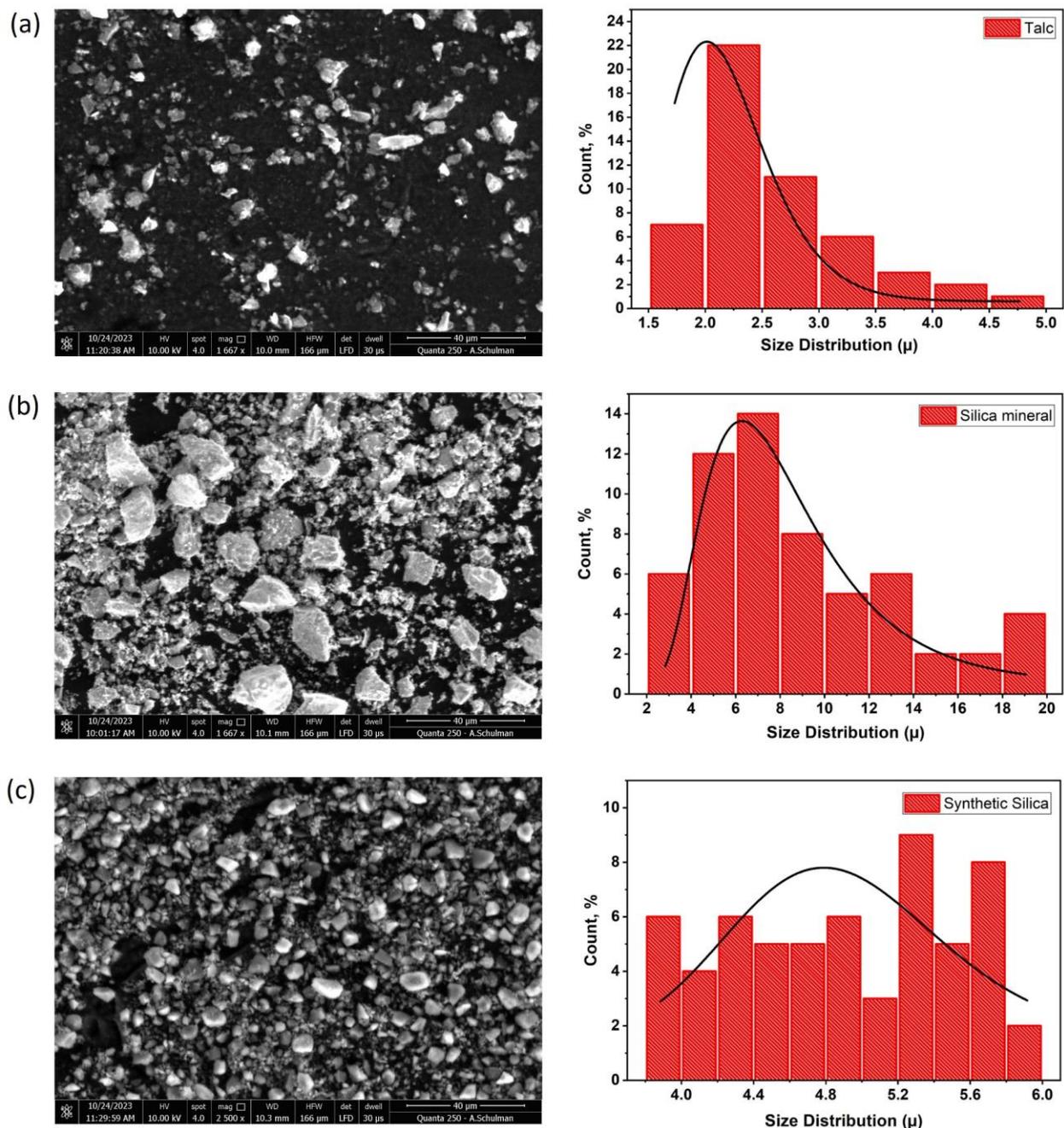
Figure 1. Infrared spectra of anti-block materials.

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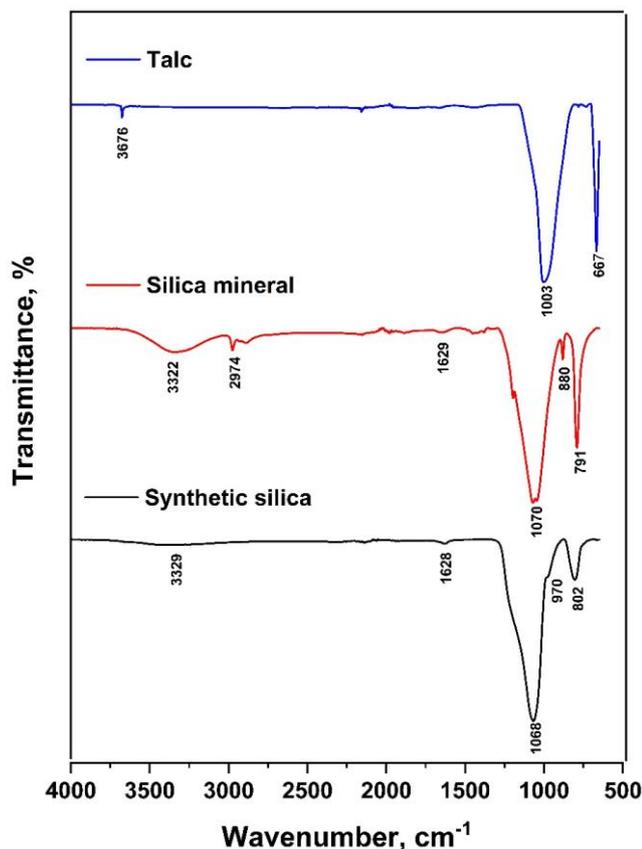
Figure 2. Infrared spectra of anti-block materials.

FTIR characterization was carried out to identify the functional groups of each anti-block material. **Figure 2.** showed the infrared spectra of anti-block materials. Talc anti-block has 3 three typical peaks at 3676, 1003, and 667 cm^{-1}, representing the Si-O tetrahedral layer and MgO/MgOH octahedral layer, respectively [15]. According to the talc structure, this functional group of talc consists of two monomer structures of silica tetrahedral and magnesia octahedral that bound each side by a silica layer [16]. From IR spectra, the talc was not found at the the O-H peak at around 3330 cm-1, which is in line with the characteristic of talc that is hydrophobic nature[17]. This feature of talc could give favorable properties such as easy blending and good dispersion with polymer [15]. IR spectra of both synthetic silica and silica mineral showed characteristic peaks at 1070 and 800 cm^{-1}, corresponding to the Si-O bond and Si-O-Si bending vibration of pure SiO_2 [18]. The peak at 970 cm-1 in synthetic silica represented the Si-O signal, and the peak at 880 in mineral silica correspond to the –OH functional group of SiO_2 [19,20]. The peak at 1629 and 3300 cm^{-1} were found in silica mineral and synthetic silica, representing OH bending vibration due to adsorbed water, and the hydrophilic nature of the material. The peak at 2974 cm^{-1} in silica mineral was a C-H stretching vibration that might come from the residual resin masterbatch [21].

Slip additive analysis on Surface Polyethylene Film

Figure 3. showed the chromatogram of additives in the slip masterbatch, which has only two peaks. The first peak at retention times 10.873 min was pyrene as the internal standard, and the peak at retention times 10.808 min was the analyte of the masterbatch slip additive. The identification of the slip additive peak was determined by GC/MS library (NIST), and the obtained mass spectra in scan mode at RT 10.808 of the analyte was similar to the mass spectra of erucamide with a qualitative of 93. The qualitative report by the GC-MS revealed that the slip masterbatch contained the slip additives with the type of erucamide. The molecular ion peak at 337, and the fragmentation pattern corresponded to the GC-MS result of erucamide reported by [22].
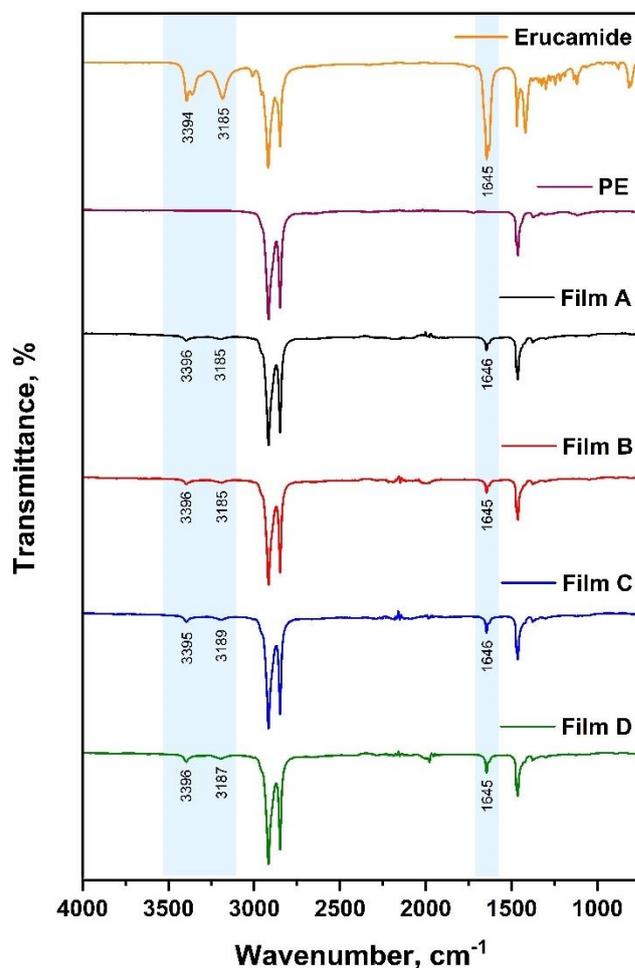
Figure 4. Infrared spectra of erucamide, polyethylene film, and formulated films.
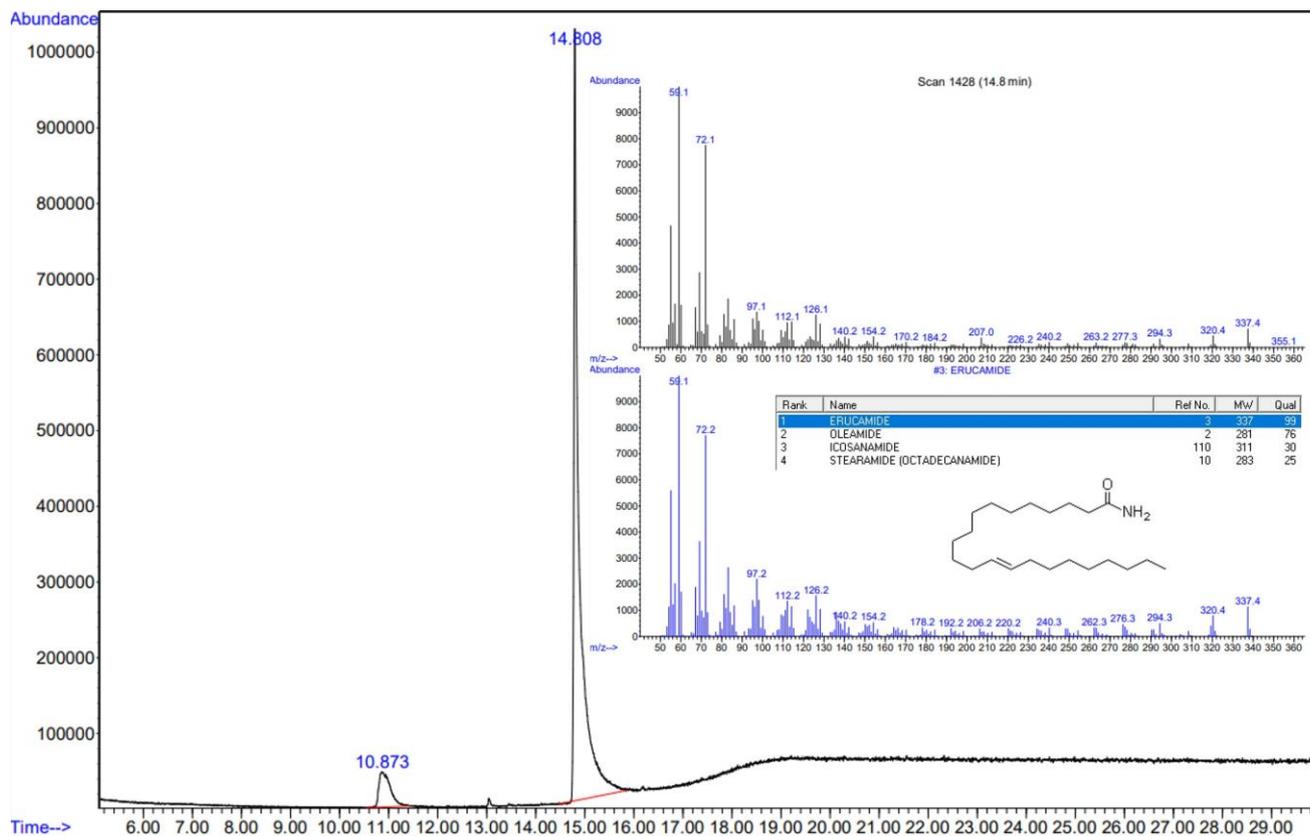
Figure 3. Gas chromatogram of slip additive and mass spectra obtained in scan mode at RT 14.8 min.

Film samples were prepared by combining slip additives with several types of anti-blocking materials to study the influence of inorganic anti-block additives on the migration behaviour of slip additives on film surfaces. The migration of slip additives on the surface was due to differences in structure between slip and polymer [9]. Erucamide, as a slip additive, has a polar hydrophilic amide group and a non-polar hydrophobic hydrocarbon chain [23]. The polar group of erucamide will migrate to the surface film, giving a layer and acting like a lubricant, and the non-polar group will stay inside the film due to its similar structure to polyethylene [24,25]. The film was stored in a humidity chamber to allow the migration process. After a period of aging the films, the relative amount of erucamide - combined with a different type of anti-block, which migrated to the film surfaces was determined by FTIR. **Figure 4** showed the polyethylene film has a specific peak at 1472-1462 cm^{-1} due to methylene scissoring [8]. The presence of erucamide has a characteristic peak at 3407 cm-1 corresponding to the N-H stretch and carbonyl stretching peak at 1645 cm^{-1} [26,27]. The erucamide content on the surface of each film formulation is shown in **Table 4**.

In this study, at the same loading erucamide additive in polyethylene carrier resin, the content of erucamide on the surface was found to be different in each used type of anti-block additive. On the first day, the erucamide content on the surface is found to be lowest on Film D, followed by Film C, Film B and Film A**.**

Table 4. Amount of Erucamide on surface film.

Aging	Erucamide conetent (ppm)			
	Film A	Film B	Film C	Film D
Day 1	621.0	588.4	488.2	470.4
Day 3	581.8	437.4	465.4	582.1
Day 7	656.2	429.2	540.0	394.6

This trend is in alignment with the silica content presence on each anti-block additive based on SEM EDX result, which the synthetic silica with higher relative silica content can more inhibit migration of erucamide than other anti-block additives. Inorganic anti-block additives such as talc, natural silica, and synthetic silica were utilized to regulate the slip migration on the surface. Compared to Film A, the erucamide content was in stable migration from day to day due to not used of anti-block that can prevent

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the migration. The O-H group of the inorganic surface can interact with erucamide additives via hydrogen bonding and reduce migration of the slip additives [28]. This interaction between silica and erucamide will develop each day, and after day 7, gave an interesting result that erucamide on Film C has higher erucamide content on the surface than Film B. This finding might be due to the used anti-block type in Film C was natural silica, which has random micro-size particles that can cause agglomeration and reduce the interaction between erucamide and silica. Talc anti-block additives could also present the polar hydroxyl group on particle edges that interacted better with the polar amide end group of erucamide than natural silica [29].

The migration of erucamide on surface Film A, Film B, Film C, and Film D after day 7 were 44%, 29%, 36%, and 26%, respectively. Film D has the lowest relative content of erucamide than other film formulations. It is noticed that anti-block material type synthetic silica is more effective in inhibiting the migration of erucamide to the film's surface than films containing talc and natural anti-block types. The lower erucamide on the surface might be adsorbed onto the silica surface or trapped within the matrix [6] .

Optical properties

Haze is identified as the scattering of light, defined as "smokiness" in the film [4]. The addition of inorganic anti-block additives in this study resulted in negative effects on optical properties. **Figure 5** showed the haze value of all film formulations vs. days. From day 1 to day 7, the haze value of each film increased, indicating there was migration of erucamide on the surface film, affecting the optical properties of each film to become hazier. The highest haze value of film formulation was Film C, which used natural silica anti-block, which has coarser and random distribution of particle size. The particle size distribution affected the haze level of the end optical properties of plastic. The finer the anti-block size, the lower the haze value [30].

From SEM EDX data, The Si content in synthetic silica anti-block was the highest compared to natural silica and talc. The high silica content of anti-block material can also result in high haze value [4]. Otherwise, Film A possessed the lowest haze value due to no anti-block additive used in film formulation. Film D used synthetic silica anti-block, which has a higher Si content than the natural silica anti-block, but interestingly, the optical properties was lower than Film C. This result indicates there is another factor besides element content that affected the optical properties of the film. From SEM Data, synthetic silica has a higher silica content, but it has a smaller and uniform particle size than natural silica. In contrast, natural silica has lower silica content and also has a higher and random

distribution particle size. A larger particle of inorganic anti-block may be more effective at avoiding blocking but commonly increased the film haze [4]. The inorganic anti-block with smaller particles can give better clarity [31]. The smaller particle size allows good dispersion and facilitates better interaction between silica and erucamide, supported by FTIR data. The good dispersion can result in better clarity even synthetic silica has higher silica content than natural silica. In contrast, the natural silica has a rough surface and random distribution of particle size that might be agglomerate. This different anti-block characteristic gave result on high haze value of Film C. From the haze result, we can conclude that the type of inorganic anti-block, which has different sizes, shapes, and silica content, affected the haze of the film [32].
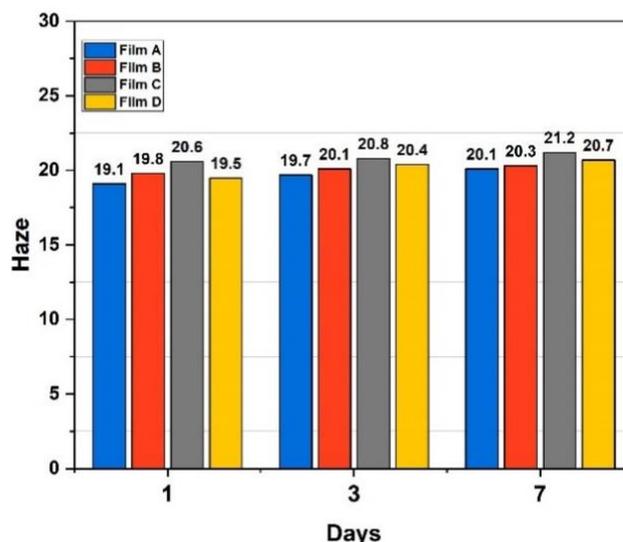
Figure 5. Haze values of formulated films during aging process.

Coefficient of friction analysis

The coefficient of friction consists of static and kinetic COF. Static COF is the force measured to begin movement, and kinetic COF is the force needed to sustain this movement. COF affects how a film slides over itself and other surfaces [33]. **Figure 6** showed the kinetic COF test result of each formulation film. The COF result of all film formulations showed that each film included in the high slip category has a below 0.200 COF value [4]. Film A, with only slip additive, possessed the lowest COF value. This slip additive has a low surface energy and reduces the adhesion between the asperities to lower the frictional properties. Film B, C, and D with anti-block addition have a higher COF value than film without anti-block [4]. The inorganic anti-block additive is used as an aid to control high medium slip [35]. This is because inorganic

additives, such as synthetic, natural silica, and Talc, can increase the total of asperities and surface roughness, which facilitate the maintain of frictional properties. The COF test result of all formulations was developed and reduced after 7 days from film extrusion [10]. The development of COF value was influenced by erucamide. At first, erucamide, after extrusion, is homogeneously dispersed in the film, and the concentration of erucamide content on the surface was low, resulting in high COF, as shown on Day 1. The erucamide belongs to a monounsaturated fatty acid with a polar primary amide group and a non-polar hydrocarbon chain with 22 carbon atoms. Because of the limitation and incompatibility of the polar group, the amide group will go to the surface and migrate. Thus, its effect on the slip concentration increased after day 1, and the COF value decreased. The migration will develop continuously to reach equilibrium, which gives the formation of a slip layer on the surface and attains minimum COF value [35].
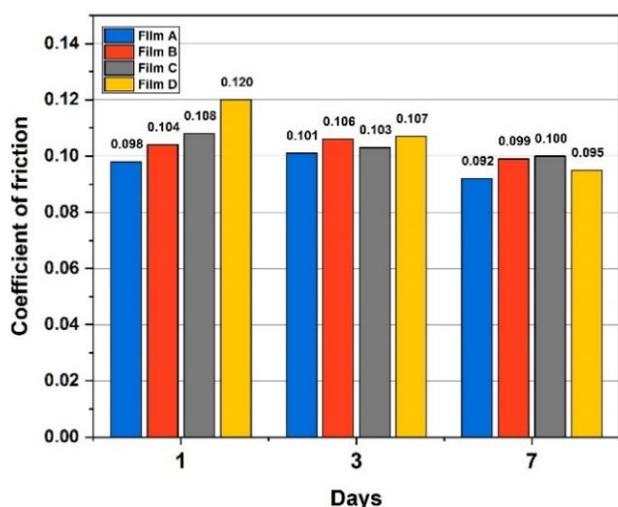
Figure 6. Coefficient of friction of all formulation film vs days.

Film B with talc anti-block additives has the same synergist trend of COF development as Film A without anti-block additives, which is higher on day 3 and reduced on day 7. In comparison, Film C and D have the same trend, in which the COF value decreased from day 1 to 7. This finding correlated with the FTIR data of anti-block which is talc that used in film B has an hydrophobic in nature while synthetic silica and mineral silica was more hydrophilic in nature. These two different characteristics of anti-block gave two trend of COF development mechanism. The polar group of Si-O

silica in synthetic silica and mineral silica can interact better to erucamide to preserve it and prevent the migration to the surface which is give the stable decrement trend from day 1 to day 7. In contrast talc with hydrophobic nature is less interact with polar group of erucamide and only can interact with the edges of surface polar group resulting continuous migration in day 3. From day 1 to day 7, Film D with natural silica anti-block content has the lowest and faster development of COF value than other inorganic additives. After day 7, the COF value of all formulation film was Film A < Film D < Film B < Film C, which in accordance with the erucamide content in surface film A > Film D > Film B > Film C. The reduced COF value after anti-block addition showed the control of slip migration by anti-block and after 7 days, for Film A, B, C and D, the COF value was decreased by 6%, 5%, 7% and 21%, respectively. The decrement of COF value for Film B, C, and D correspond to silica content in anti-block, which gradually increased from talc, natural silica, to synthetic silica. The silica content in anti-block can adsorbed slip additive and reduce availability in film surface [30]. The higher silica content was contributed to faster slip development of COF value, but the end properties of friction of all film formulations were not aligned with silica content, indicating another factor that affected the end friction properties. The use of anti-block additives in film can also act as a micro bump and roughness addition on the film surface, which the silica particles stick out of the film surface and give a space bar between two adjacent films to minimize film-to-film contact [7,30,36].

From SEM data showed the shape characteristic and size of the anti-block used were different. Film D has synthetic silica type, which has the highest silica content, smooth shape, and has a size distribution of 4-5 μm. The balance of anti-block characteristics and the strength to control migration speed in Film D resulted in the lowest and faster COF development value than other used anti-blocks in this experiment. On the other hand, Film C used natural silica, which has a rough shape and higher and random distribution micro size particle that gave the highest COF value even than talc, which has a lower silica content [30]. After day 7, film B possessed a lower COF value than Film C due to the smaller particle size of talc result in lower COF value. The silica content, size, and shape of the anti-block shape determine the end friction properties of the film. The illustration of the anti-block and slip additive relation in the film was shown in **Figure 7**.

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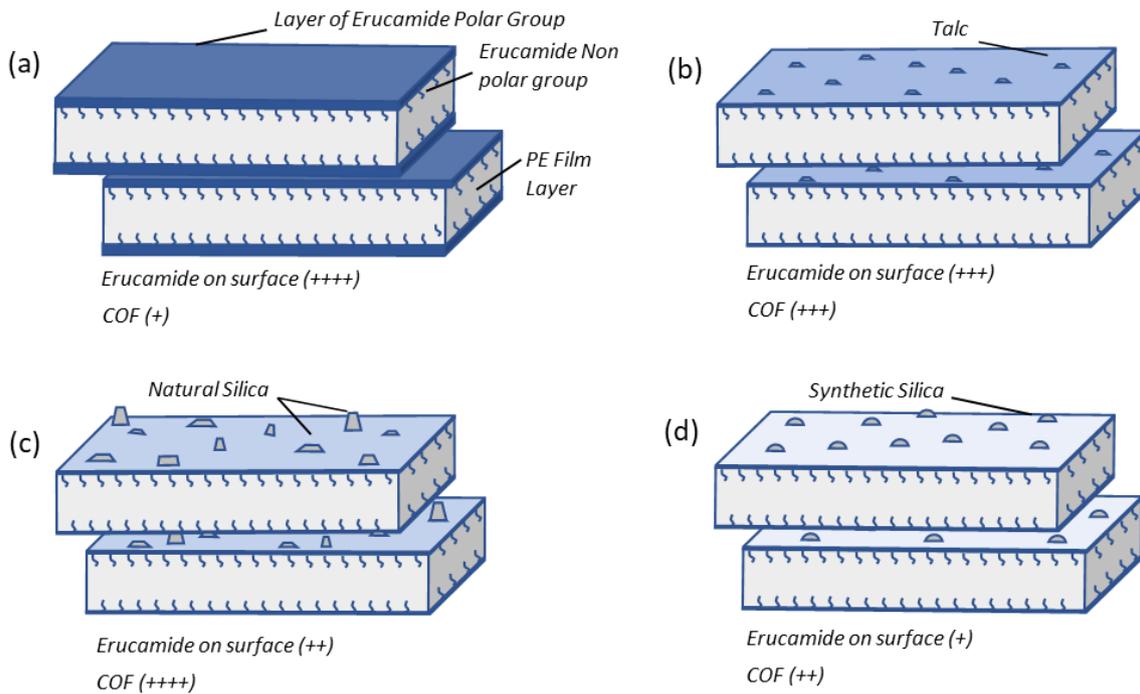
Figure 7. Illustration of surface character of (a) Film A, (b) Film B, (c) Film C, and (d) Film D. The darker the color resembling the higher erucamide content.
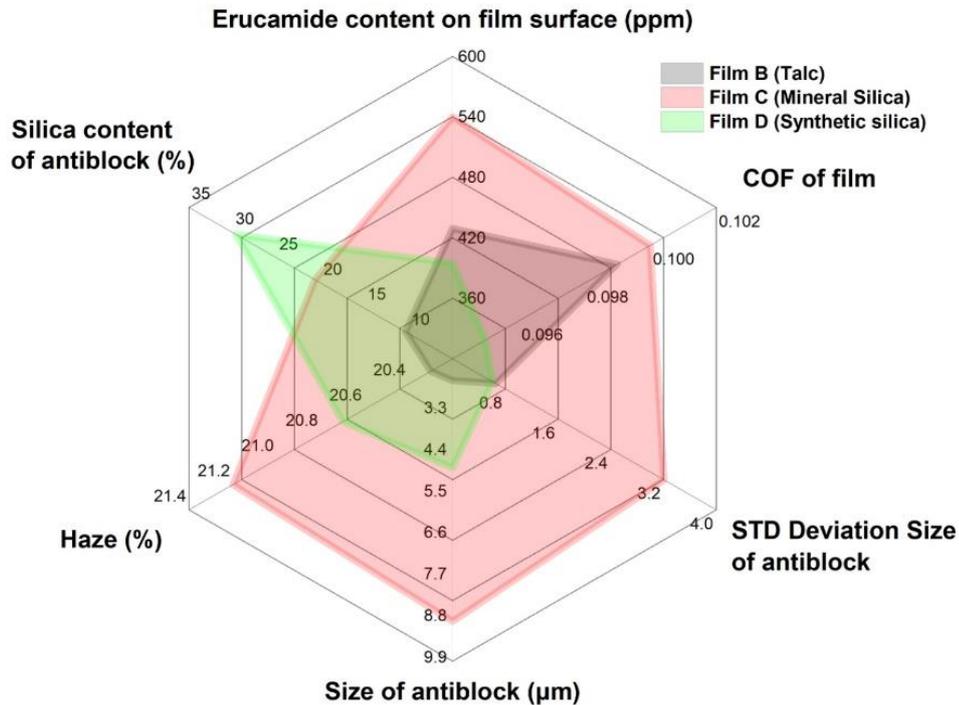
Figure 8. Characteristics of Film B, Film C, and Film D (based on haze, erucamide content on film surface, and COF of the 7 days of aging)

The characteristics of Film B, Film C, and Film D after 7 days of aging were illustrated in **Figure 8**. From the figure, it can be concluded that large random and particle sizes, as seen on mineral silica in Film C, can have a negative effect on polyethylene film performance due to the lowest result in optical properties, friction properties, and migration control of the film. In film D, the characteristic of synthetic silica with a higher silica content can regulate the lowest migration of erucamide, resulting in good friction properties, but it is hazier in optical. In comparison, Film B with talc anti-block has superior optical properties but has lower migration control characteristics, resulting in

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moderate friction properties compared to other films.

CONCLUSION

In this study, anti-block additive to control the migration of slip was carried out by FTIR analysis, SEM-EDX, COF, and Haze characterization to evaluate the development properties. Addition of synthetic silica anti-block to PE film containing slip additive could reduce the migration of erucamide content on PE surfaces up to 39.9% lower than that of film without anti-block, giving better COF properties than other anti-block additives. For the optical properties, Talc and synthetic silica showed a low optical property with a haze value of 20.3% and 20.7%, respectively. This value indicates that the clarity of polyethylene film was affected by the silica content, shape, and particle size of each anti-block used. This study of slip migration can provide recommendation to the flexible-food packaging industry to formulate the film structure to avoid the excess of slip additives on the film packaging surface.

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