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MECHANICAL PROPERTIES OF ETHYLENE VINYL
ACETATE COMPOSITE SHEET FOR MASS BACK



Mr.Vuttipol Boonjungmongkol

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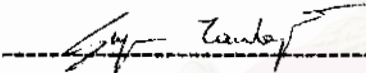
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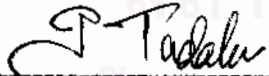
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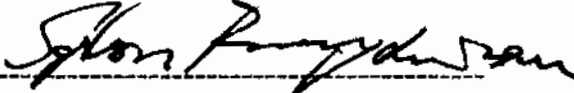
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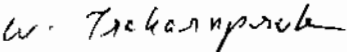
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ศึกษาพอลิเมอร์คอมพอสิตของอีวีเอ 2 ชนิด, แอลดีพีอี และสารเพิ่มเนื้อได้แก่ แคลเซียมคาร์บอเนต เคโอลิน ทัลค์ และแบเรียมซัลเฟต โดยผสมอีวีเอ แอลดีพีอี และสารเพิ่มเนื้อในอัตราส่วนต่างๆ ดังนี้ 30/5/65, 25/5/70 และ 20/5/75 ด้วยเครื่องผสม 2 ลูกกลิ้ง ที่ความหนา 2 มิลลิเมตร โดยศึกษาผลของปริมาณและชนิดของสารเพิ่มเนื้อ รวมทั้งปริมาณของอีวีเอในอีวีเอ ที่มีผลต่อสมบัติเชิงกลของพอลิเมอร์คอมพอสิต สมบัติเชิงกลของพอลิเมอร์คอมพอสิตที่ศึกษาได้แก่ ค่าการทนแรงดึง ค่าเปอร์เซ็นต์การยืดและค่าความแข็งที่ผิวแบบชอร์เอ โดยค่าการทนแรงดึง และค่าเปอร์เซ็นต์การยืดจะทำการทดสอบ 2 แนวคือแนวตามเครื่อง และแนวขวางเครื่อง จากการทดสอบค่าสมบัติเชิงกลพบว่า เมื่อปริมาณของสารเพิ่มเนื้อเพิ่มขึ้น จาก 65 ถึง 75 เปอร์เซ็นต์ ค่าการทนแรงดึงและค่าเปอร์เซ็นต์การยืดของพอลิเมอร์คอมพอสิตลดลง ในขณะที่ค่าความแข็งที่ผิวเพิ่มขึ้น และเมื่อปริมาณอีวีเอเพิ่มขึ้นจาก 18 เป็น 22 เปอร์เซ็นต์ ส่งผลให้ค่าการทนแรงดึงและค่าความแข็งที่ผิวลดลง แต่ค่าเปอร์เซ็นต์การยืดจะเพิ่มขึ้น

อัตราส่วนที่เหมาะสมของพอลิเมอร์คอมพอสิตสำหรับทำแผ่นรองพื้นคือ อีวีเอ เกรด N8038/แอลดีพีอี/ทัลค์, 30/5/65, มีค่าการทนแรงดึงแนวตามเครื่อง แนวขวางเครื่อง ค่าเปอร์เซ็นต์การยืดแนวตามเครื่อง แนวขวางเครื่อง และค่าความแข็งที่ผิวแบบชอร์เอ คือ 441.3 psi, 418.3 psi, 459.0%, 438.3% และ 83.3 ตามลำดับ ส่วนพลาสติกเชิงพาณิชย์มีค่าการทนแรงดึงแนวตามเครื่อง, แนวขวางเครื่อง, ค่าเปอร์เซ็นต์การยืดแนวตามเครื่อง, แนวขวางเครื่อง และค่าความแข็งที่ผิวแบบชอร์เอ คือ >300 psi, >200 psi, >400%, >350% และ 75-85 ตามลำดับ สมบัติเชิงกลของพอลิเมอร์คอมพอสิตที่เตรียมได้นี้อยู่ในช่วงที่ยอมรับได้ของพลาสติกเชิงพาณิชย์ และมีราคาที่ถูกมาก

หลักสูตร
สาขาวิชา ปิโตรเคมีและวิทยาศาสตร์พอลิเมอร์.....
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VUTTIPOL BOONJUNG MONGKOL : MECHANICAL PROPERTIES OF ETHYLENE
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The polymer composites of EVA, LDPE and fillers (CaCO_3 , kaolin, talc and BaSO_4) were investigated. The EVA, LDPE and fillers in various compositions such as 30/5/65, 25/5/70 and 20/5/75 were mixed in two-roll mill to make 2 millimeters thickness sheets. The study revealed the effect of type and filler content, VA content on mechanical properties of polymer composites. The mechanical properties were determined by using tensile testing and hardness Shore A testing. The tensile strength and ultimate elongation were tested in 2 directions; machine direction (MD) and transverse direction (TD). The tensile strength and ultimate elongation of polymer composite decreased while their hardness increased when filler contents were increased from 65 to 75%. The increasing of VA contents from 18 to 22% caused the tensile strength and hardness to decrease but the ultimate elongation increased.


The best composition of polymer composite for mass back was EVA(N8038)/LDPE/talc at 30/5/65. The tensile strength in MD and TD, the ultimate elongation in MD and TD and the hardness Shore A were 441.3 psi, 418.3 psi, 459.0%, 438.3% and 83.3, respectively for the prepared polymer composites, meanwhile, the corresponding values of the commercial resins in the above criteria were >300 psi, >200 psi, >400%, >350% and 75-85, respectively. It was noted that the mechanical properties of prepared polymer composites were in acceptable range of commercial resin. Moreover, this polymer composite was very low cost.

Program _____

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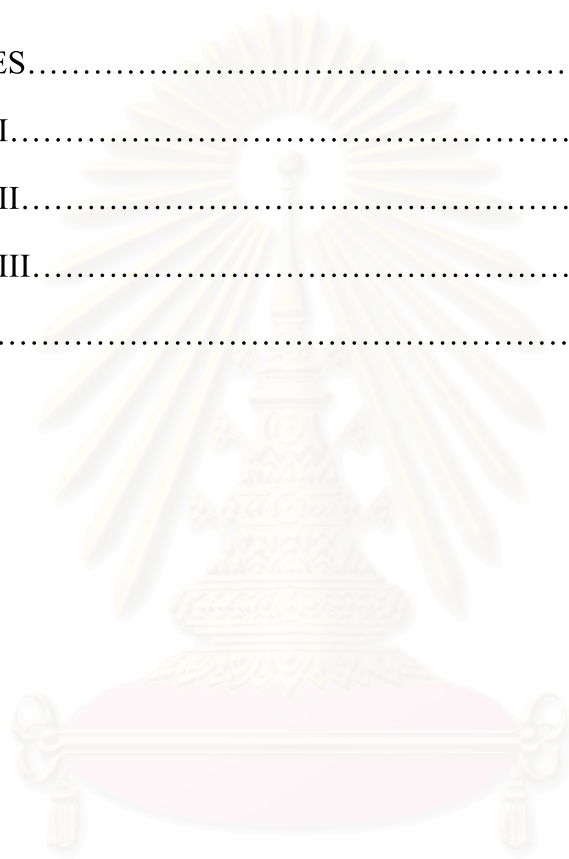
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LIST OF ABBREVIATIONS

APEN	Alkyl di-hydrogen phosphate ester
ASTM	American Society for Testing and Materials
EL	Elongation
esd	Equivalent spherical diameter
EVA	Ethylene vinyl acetate copolymer
HD	Hardness
ISO	International Standards Organization
LDPE	Polyethylene
MD	Machine direction
n_D	Refractive index
psi	Pounds per square inch
S.G.	Specific gravity
SAE	Society of Automotive Engineer
TD	Transverse direction
TPE	Thermoplastic elastomer
TS	Tensile strength
VA	Vinyl acetate

CHAPTER I

INTRODUCTION

1.1 State of Problem

It is well known in the automobile and truck industry to use fully carpeted floor covering to create a plush, aesthetically pleasing appearance for passenger compartment. However, fully carpeted floor coverings are much more expensive than corresponding synthetic 'polymeric based' floor coverings because of the raw material cost of the carpet. Further, fully carpeted floor coverings rapidly lose their showroom appearance due to their exposure to road dirt, mud, solar radiation and salt, for example. Therefore, a floor covering which utilized less carpet than typical carpeted floor coverings, which also creates similar plush, aesthetically pleasing interior appearance and which would retain its showroom appearance, would be advantage.

1.2 Auto Carpet Backing

Auto carpet backing is extruded direct onto the carpet back. The carpet is then shaped in a double-faced thermoform mold, trimmed, and shipped to the auto assemble plants as a one-piece form-fitting unit, which is easily installed in the car without trimming, seaming, or cutting (see Figure 1.1).

In the part, auto carpet backing used poly(vinyl chloride) based resin as floor mat. Nowadays, many countries forbid the use of poly(vinyl chloride). Therefore, other polymers have been used to substitute poly(vinyl chloride). Ethylene vinyl acetate is one of them.



Figure 1.1 One-piece, form-fitting carpet units retain their shape which being flexible and easy to handle on the production line

1.3 Purpose of the Research

To improve the mechanical properties of ethylene vinyl acetate composite resins by adding various types of fillers.

1.4 Scope of the Research

Composites were prepared by mixing ethylene vinyl acetate copolymer, low-density polyethylene, and fillers in various ratios with two-roll mill for 2 millimeters composite sheets.

The mechanical property testing, which are important for composite such as tensile strength, ultimate elongation and hardness would be carried out.

CHAPTER II

THEORY AND LITERATURE REVIEWS

2.1 Thermoplastic Elastomer

A thermoplastic elastomer (TPE) is a rubbery material which can be processed in the molten state as a thermoplastic and which has many of the performance characteristics of a thermoset rubber [1]. By definition, a TPE is an elastomer because of its elastic properties, such as its flexibility, in case of distortion under applied load and its recovery of most of its original shape after removal of an externally applied stress. A TPE is also a thermoplastic because it is fluid at temperatures above its melting point and can be processed on thermoplastic extrusion and molding equipment [2]. Unlike a thermoset rubber, a TPE can be recycled to recovery scrap from processing operation and, in many cases, to allow material reuse at the end of the useful life of a TPE article [3].

Commercial TPE covers a hardness range from 30 Shore A up to 75 Shore D, with the number of grades available increasing progressively with hardness. With increasing hardness, the rubber-like properties of a TPE decrease progressively, and the plastic properties increase. TPE have useful temperature ranges from as low as -70 to as high as 171°C and are capable of extended service in a broad spectrum of environments. Thus they are viable alternatives for uses which heretofore have been the specific advantage of a thermoset rubber.

Table 2.1 Comparative hardness scales for plastic [4]

Brinnell	Rockwell		Shore		Typical products
	M	R	D	A	
25	100				Very hard
16	80				
12	70	100	90		
10	65	97	86		
9	63	96	83		Semiflexible
8	60	93	80		
7	57	90	77		
6	54	88	74		Flexible
5	50	85	70		
4	45		65		
3	40		60	98	Golf ball cover
2	32		55	96	
1.5	28		50	94	
1	23		42	90	Faucet washer
0.8	20		38	88	
0.6	17		35	85	
0.5	15		30	80	
			60		The tread
			50		Inner tube
			35		Rubber
			10		

Injection molding, blow molding, extrusion, thermoforming can also be processed TPE and heat welding is extremely rapid and highly economical. All of these methods are unavailable for the fabrication of thermoset rubber articles. The suitability of these processing methods is derived from the thermoplastic nature of a TPE [5].

The practical application of TPE is virtually as broad and intensive as those of conventional thermoset rubbers. A major exception is that of pneumatic tyre, for which no TPE has been found suitable. Suitable TPE applications include non-tyre, automotive, major and minor household applications, building construction, shoes, electrical insulation and jacketing, sheeting, food and beverage contact, and health care. The several types of TPE are treated in detail in the articles that follow [6].

2.2 Polymer Matrix

Commercially, polymers are almost always mixed together with various additives, which can be either monomeric or polymeric and are in the solid, liquid, or gaseous state. Their presence is intentional; it is aimed at achieving specific desired properties of the end product or ease of processing. Examples of additives used in plastics are fillers, reinforcing agents, foaming agent, plasticizer, stabilizers (thermal as well as environmental), lubricants, and pigments [7].

From the point of view of properties and end use, we can categorize pure and compounded polymers as plastics, elastomers, and fibers.

2.2.1 Ethylene Vinyl Acetate Copolymer

Ethylene vinyl acetate copolymer (EVA) is made by the copolymerization of ethylene monomer and vinyl acetate (VA) monomer. Most often, this free radical polymerization, initiated by either a peroxide or perester, is done in a conventional stirred autoclave or high-pressure tubular reactor [6,8-9].

EVA included a broad range of thermoplastic materials with VA contents from below 5 to 50%. Products made from EVA, depending on VA content, range from thermoplastic to elastomer. EVA is mainly recognized for their flexibility and toughness (even at low temperatures), adhesion characteristics, and stress-crack resistance. Compared to LDPE, EVA is more polar and less crystalline. The resins are used for injection and blow molding virtually all extrusion process, and they are blended and compounded with other materials.

The VA monomer results in basically LDPE-like resins with randomly distributed, pendant acetoxy groups. VA and ethylene monomers have very similar reactivity ratio; therefore, the pendant acetoxy groups in the resulting copolymer are randomly distributed.

Two main characteristics of EVA are controlled in the polymerization process: crystallinity and molecular weight.

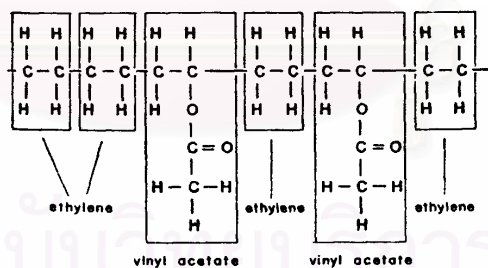


Figure 2.1 Ethylene vinyl acetate copolymer

Unlike with LDPE polymerization, increasing density of EVA does not correspond to an increase in crystallinity. Rather, as VA content increases, density also increases, but crystallinity and its related properties decrease. Thus,

as VA content increases, the low-temperature flexibility, stress-crack resistance, and impact strength clearly improve. However, there is a decrease in softening/sealing temperature and barrier properties. Above 50% VA content, EVA is totally amorphous.

EVA is used in packaging film, which is the largest single market (over 60% of total 1988 sales). Adhesive, carpet backing, and wax based coating, are the second largest EVA market (about 20% in 1988). It is made from copolymers, which typically contained 18 to 30% VA. Wire/cable and color-compounded are the third largest category (6%). Molding and extrusion applications are the fourth largest market (5%) for EVA.

2.2.2 Low-Density Polyethylene

Polyethylene has a wide range and combination of desirable properties. Its very low T_g of about -120°C , and moderate high T_m of about $105 - 115^{\circ}\text{C}$ give it flexibility and utility over a wide temperature range. It has crystallinity with a good combination of strength, impact resistance, flexibility, and melt-flow behavior over a range of molecular weight. The alkane structure of polyethylene imparts good solvent, chemical, thermal, oxidation, water, and electrical resistance. Commercial LDPE have number average molecular weights in the range 20,000 - 100,000. A wide range of fabrication techniques often refers to as low-density polyethylene (LDPE). LDPE is high branched (both long and short), low crystallinity (40–60%) and density ($0.91\text{-}0.93\text{ g/cm}^3$). LDPE find a wide range of applications as flexible plastic. About 60% of all LDPE is produced as extruded film and sheet, mostly for packaging and household uses (bags, pouches and wrap for food, garments and dry cleaning,

trash), but also for agricultural and construction applications (greenhouse, tank liners, moisture, and protective barriers). Injection molding of toys and house wares accounts for another 10-15%. About 10% or more of the LDPE are used in wire and cable insulation for power and communication transmission and hot melt coating (on paper, metal foil and other plastic films). Other uses of LDPE include blow molding bottles and containers, pipe and tubing for agricultural irrigation [6,10].

2.3 Principles of Fillers

Fillers can be divided into two categories.

a) Insert or extend filler.

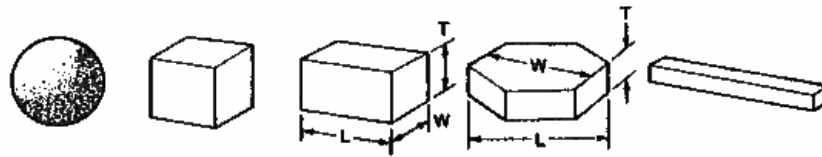
The objective of filling polymers with inert fillers is that the composite can be made at a lower cost than the unfilled materials. Commonly used materials are calcium carbonate, clay and talc.

b) Active or reinforcing filler.

The reinforcement of polymers increases mechanical properties. Reinforcement depends on a fine particle size and high aspect ratio in the filled polymer. Examples of this type are carbon black and fine particle silica [10].

2.3.1 Properties of Fillers

Filler selection is primarily determined by the particle size distribution, the particle shape, and, as a consequence of both, the manner in which the particles pack together. First, an examination of the particles as the primary unit of the filler is required.

Table 2.2 Particle characteristics

Particle Class	Sphere	Cube	Block	Flake	Fiber
Descriptor	spheroidal	cubic	tabular	platy	acicular
		prismatic	prismatic	flaky	elongated
		rhombohedral	pinacoid		fibrous
			irregular		
Shape ratios;					
length (L)	1	~ 1	1.4 - 4	1	1
width (W)	1	~ 1	1	< 1	< 1/10
thickness (T)	1	~ 1	1 - < 1	1/4 - 1/100	< 1/100
Sedimentation					
diameter	1	esd esd	esd	esd	esd
Surface area					
equivalence	1	1.24	1.26 - 1.5	1.5 - 9.9	1.87 for 1/100 2.3 for 1/20
Examples	glass spheres microspheres	calcite feldspar	calcite feldspar silica barite nephelite	kaolin mica talc graphite hydrous alumina	wallastonite tremolite wood flour

A general classification of filler particles is presented in Table 2.2. The classes are based on a somewhat arbitrary classification according to surface area. This classification is based on two primary properties of fillers; particle size and surface area, both of which are directly measurable and serve as a basis for systematizing filler functions. Most filler, and those commonly used,

are minerals, which are ground rock or ores otherwise processed to obtain the material in particulate form. As such, they do not cleave, or fracture uniformly and their particles are generally irregular shapes. Certain fabricated fillers, such as glass spheroids, precipitated silicates, and calcium carbonates, will have more precise forms [11-12].

Hence fillers are today employed to obtain any, or a combination, of the following effect [13].

1. Add rigidity, stiffness or hardness.
2. Regulate thermal expansion and shrinkage.
3. Improve heat resistance.
4. Improve or regulate electrical characteristics.
5. Increase strength and reduce creep.
6. Modify rheological properties (flow, thixotropy and body).
7. Aid processability (lubrication, flow, mixing, dispersion, etc.).
8. Modify appearance (opacity, color and texture).
9. Alter density and bulk.
10. Lower cost.

2.3.2 Effect of Fillers on the Properties of Polymers

Many mineral fillers are used in various polymer systems to enhance physical or mechanical properties. There is a lot of scientific literatures showing the effect of filler on polymer properties. Mechanical properties of filled polymer are indeed dependent on many parameters: matrix properties, particle characteristics (nature, size, shape, and size distribution), constituent volume fraction and matrix-filler interaction [14].

2.3.2.1 Tensile Strength

Tensile yield stress (σ_{yc}) depends on the filler content and mechanical properties of the filler constituents, shape and space packing of the filler particles, and degree of interfacial adhesion. The tensile yield stress of compounds is generally reduced with increasing filler volume fraction. Filler reduces the effective cross sectional area of the matrix and hence reduce its load bearing capacity. This leads to an increase in internal stress, at any given external loading, compared with the unfilled matrix. Stress concentrations caused by the filler also contribute to the internal stress [15-16]. Microvoid deformations occur around particles facilitates damage of the material at lower external loads, compared with unfilled matrix. The theory of Nicolais and Nicodemo predicts the following relationship [13,17].

$$\sigma_c = \sigma_m (1 - a\Phi^b) \dots\dots\dots 2.1$$

where Φ is the volume fraction of filler.

σ_c , σ_m is the filled composite and the matrix strength,
respectively.

a is related to stress concentrations.

b is related to geometry of the filler.

2.3.2.2 Elongation

Fillers tend to decrease the tensile elongation of a material. The decrease of elongation at break (ϵ_b) with rigid filler content is illustrated in Figure 2.2.

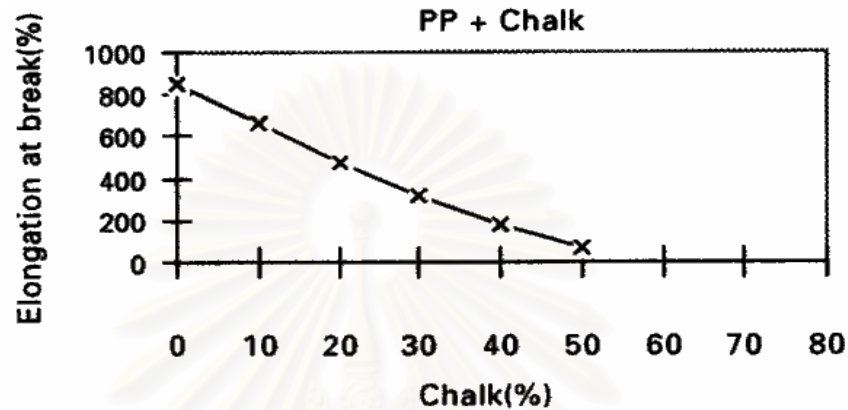


Figure 2.2 Tensile elongation at break of PP filled with various amounts of Chalk [18]

When a tensile load is applied, polymer composites begin to deform. Filling invariably reduces deformation of the samples. The drop in deformation due to the presence of the filler may be explained by a smaller concentration of polymer matrix in the same section and volume. In this case, relative tensile elongation can be determined from the equation 2.2 [19].

$$\epsilon_c / \epsilon_m = 1 - \Phi^{1/3} \quad \dots\dots\dots 2.2$$

- where
- ϵ_c is the elongation of the composite.
 - ϵ_m is the elongation of the matrix.
 - Φ is the filler volume fraction.

2.3.3 Description of the Individual Filler

2.3.3.1 Calcium Carbonate

The next most abundant naturally occurring element is calcium, which is found mainly as calcium carbonate, the principal constituent of chalk, limestone and marble. These materials have been largely derived from the consolidation of deposits of minute marine organisms during the formation of the earth's crust. Chalk deposits have resulted from soft compression, limestone rocks from hard compression and finally metamorphism has given marble [1,11].

CaCO_3 fillers have high volume in plastics. CaCO_3 can exist in two crystalline forms, calcite and aragonite that have different specific gravity (S.G) and refractive index (n_D): aragonite has S.G. 2.93 and n_D 1.53, while calcite has S.G 2.71 and n_D 1.48. Calcite is formed by crystallization of CaCO_3 at temperature below 30°C and occurs in limestone, and chalk. Aragonite is obtained by crystallization at higher temperature and is major constituent of seashells. Precipitated CaCO_3 may consist of a mixture of two crystalline forms (Table 2.3).

High loading levels of CaCO_3 lead to increased stiffness and inferior mechanical properties such as tensile strength and elongation. The factors that appear to produce the losses in physical strength include particle shape, size and the lack of interaction at the filler and matrix interface [20].

2.3.3.2 Kaolin

Kaolin, the end product of the weathering of granite and feldspar, is found in many parts of the earth, but rarely in the required degree of purity.

Kaolin is also known under the name porcelain earth and china clay or clay. It consists of primary and secondary kaolinites. In their lamellae structure, the primary kaolinites have a ratio of length to thickness of 10:1, the crystalline form of the individual lamellae is hexagonal.

Kaolin is hydrated aluminum silicate possessing a clearly determinable crystal lattice with a plate-like, hexagonal structure. The typical chemical composition of a hard kaolin with the molecular formula $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ is Al_2O_3 (38.8%), SiO_2 (45.4%), MgO (0.2%), K_2O (0.97%), Na_2O (0.07%), FeO (variable) (Table 2.3).

Kaolin generally possesses a high degree of whiteness, is nonconductor of electricity, highly resistant to chemicals and has good resistance even towards strong acids. Hard kaolin has a slightly acidic reaction with a pH of 5.0 to 5.6 [21].

2.3.3.3 Talc

Talc or hydrous magnesium silicate in its purer forms occurs as the alteration products of magnesium carbonate rock by the natural action of hydrothermal solutions. These purer forms are called steatite or steatitic talc. Commercially important deposits are also found in altered ultra basic igneous rock. The less pure talc or soapstone is contaminated with serpentine, chlorite, termolite, dolomites and magnesium. The mineral talc is a hydrated magnesium silicate with the theoretical formula: $3\text{MgO} \cdot 4\text{SiO}_2 \cdot \text{H}_2\text{O}$, or 31.7% MgO , 63.5% SiO_2 , and 4.8% H_2O . Talc occurs in four particle shapes: fibrous, lamellae, needle shaped and modular (steatitic): however, only the lamellae form is used in commercial applications (Table 2.3).

Talc is utilized as medium filler of average whiteness in thermosetting resins and thermoplastics where improvements in electrical insulation, heat and moisture resistance, chemical inertness and good machinability of moldings is required. Talc has a low absorption rate and because of its platelike structure certain grades can improve flexural properties of molding [21].

2.3.3.4 Barium Sulfate

Barytes or heavy spar (BaSO_4) is the chief natural source of barium sulfate, which frequently found in association with metallic sulfite ores often occurring as well as developed orthorhombic crystals resembling. Heavy spar are distinguished by a relatively high density of 4.3 to 4.6 g/cm^3 and Moh hardness of 2.5 to 3.5. The relatively expensive barium sulfate possesses high reflectivity (Table 2.3).

The mineral is converted to the sulfide by calcination with coal or coke and then to the chloride by treatment with hydrochloric acid. Sodium sulfate is added and blanc fixe or precipitated barium sulfate is formed. Blanc fixe is also a by-product in the manufacture of hydrogen peroxide.

Barium sulfate is obtained in dry ground or water ground grades as off white or white powders [22].

Table 2.3 Properties of mineral powders

Name	Particle shape	Density (g/cm ³)	Hardness (Moh)	Refractive index	pH
Barium sulfate	granular crystals	4.2-4.5	2.5-4.5	1.64	4-5
Calcium carbonate (nat.)	crystalline	2.7-2.71	3.0	1.49-1.66	9-10
Calcium carbonate (pptd.)	uniform crystals	2.65-2.7	3.0	1.49	10
China clay	platelike	2.6-2.63	2.0-2.5	1.56	5-6
Clay (Kaolin)	platelike	2.5-2.6	1-2	1.56	4-8
Clay (calcined)	platelike	2.5-2.63	7.0	-	5.8-6.3
Talc	platy, acicular	2.7-3.0	1-2	1.54-1.57	9-10

2.4 Literature Reviews

Schofield, Hust, Liaum, and Rathon [23] studied the effect of magnesium hydroxide (flame retardant filler) for EVA which are widely used in cable applications. Magnesium hydroxide (Premier Periclase DP393) was used as a flame retardant loading of 60% w/w in an EVA copolymer with 18% vinyl acetate. The particular modifiers used were stearic acid (converts to ammonium stearate, AS) and gamma-aminopropyltriethoxysilane (aPS). The effect of surface-treated magnesium hydroxide on the processing, mechanical and flame retardant properties were determined, together with characterization of the interaction between filler and polymer. These studies have shown that, relative to the untreated filler, the use of AS coating leads to a decrease in tensile strength to a limiting value at monolayer coverage, but an increase in elongation at break, which reaches a maximum at monolayer coverage.

However, the aPS treatment enhanced both tensile strength and elongation: relative to untreated filler, when used at levels in excess of monolayer. Characterization studies suggested that this effect was related to the silane treatment which promoting the increase of filler-matrix adhesion, and at levels greater than monolayer, promoting formation of a thick interphase and possible crosslinking of the matrix.

Tjong, Li, and Cheung [24] prepared CaCO_3 /polypropylene composites by injection molding. The morphology, static tensile, and impact properties of these composites were investigated in this study. Scanning electron microscopic (SEM) observations revealed that the beta-spherulites of the polymer matrix of the composites exhibited curve lamellae and shelf-like structures. The fillers were found to disperse within the inter-lamellae spacing of the beta-PP composite containing 10% CaCO_3 addition. However, the filler particles tended to link together to form larger aggregates when the filler content reaches 20%. Static tensile measurements showed that the elastic modulus of the composites increases with increasing filler content but the yield strength decreases with increasing filler addition. The falling weight Charpy impact test indicated that the beta-PP polymer exhibits the highest critical strain energy release rate (G_c) value. However, there was a drastic drop in G_c of beta-PP composites with increasing filler content. The results are discussed and explained in terms of material morphology.

Mishra, Perumal, and Naisk [25] studied the effect of mechanical properties of poly(vinyl chloride)/ethylene vinyl acetate (PVC/EVA) polymer blends filled with stearic acid coated CaCO_3 (Forcal-S). Impact strength and stiffness of the composites are optimized by varying EVA and filler (Forcal-S)

contents. The results show that the stiffness of the filled PVC is a function of the concentration of the filler. The impact strength of the PVC/EVA blend depends only on the concentration of EVA. The tensile strength of the filled composites depends strongly on the degree of the interfacial bond developed between the polymer and the filler. The interfacial bond strength depends on the effectiveness of the coupling agents. The inherent properties of filled PVC/EVA blends depend on the concentration of the filler and strength of the polymer-filler interface.

Mitsubishi [26] mixed CaCO_3 of various particle shapes (sphere, cube and needle) treated with alkyl di-hydrogen phosphate ester (APEN, $\text{C}_n\text{H}_{2n}+10\text{PO}(\text{OH})_2$, $n=1, 4, 8, 10, 12$) and PP in a two-roll mill. The effect of carbon number of APEN on the mechanical properties (tensile elongation, tensile strength, impact strength, yield strength) and adhesion between PP matrix and various CaCO_3 particles and the effect of CaCO_3 shape on the storage modulus of elongated PP/ CaCO_3 have been investigated.

The tensile elongation, impact strength and adhesion of PP- CaCO_3 interface increased with carbon number of APEN (n less than or equal to 10) for needle and cube types, but the dependence of tensile properties on APEN carbon number is not clearly recognized for the sphere type.

The relative storage modulus of elongated PP/ CaCO_3 , $E\text{-c(d)}/E\text{-r(d)}$ ($E\text{-c(d)}$ -modulus of elongated PP/ CaCO_3 (untreated); $E\text{-r(d)}$ -modulus of elongated PP matrix) could sufficiently be described with a modified Halpin-Tsai equation, which contains the factors of (i) polymer matrix orientation, (ii) particle effect (content, modulus, aspect ratio), and (iii) void volume at polymer-filler particle interface.

Wang [27] investigated the improvement of the mechanical properties of PP using some elastomers and fillers. Both the mineral additives and polymer additives have affected the impact properties and tensile strength. There are also some changes in the thermal properties. To improve the interfacial adhesion, some low molecular weights of polymers are added to assist the dispersion of the fillers and the uniformity of the various polymers with PP. The addition of LDPE, HDPE, or the styrene-butylene-styrene block copolymer (SBS) can improve the impact properties of PP. The propylene-ethylene copolymer has a more pronounced effect than does the physical blending of PP with PE. It is found that CaCO_3 can be used to reinforce PP resin. The EVA has an effect on the printing properties of the PP.

Roberts [28] revealed in United States Patent No. 5,439,725 for the production of floor mat for an overland vehicle. Wherein the carpet has a predetermined surface texture covering the composite sheet (0.080 inches thickness). Floor mat was produced from mixture of poly(vinyl chloride), plasticizers, stabilizers and calcium carbonate as filler in order to reduce cost.

Martinezpardo, Zuazua, Hernandez, Cardosa, Montiel, and Vazquez [29] investigated structure-properties relationship of EVA/LDPE blend using DSC, TGA and WAXS techniques to correlate structure modifications with the changes in properties for all samples. Vicat softening temperature and hardness of specimens were increased while elongation at break was decreased with increasing of LDPE.

CHAPTER III

EXPERIMENTAL

3.1 Raw Materials

3.1.1 Ethylene Vinyl Acetate Copolymer

Commercial grade of EVA, N8038 (18% VA) and N8045 (22% VA), used in this study were supplied by Thai Petrochemical Industry Public Company Limited. Typical data of EVA grade N8038 and N8045 are shown in Appendix I.

3.1.2 Low-Density Polyethylene

Typical data of commercial grade of LDPE, JJ4324 from Thai Petrochemical Industry Public Company Limited are shown in Appendix I.

3.1.3 Calcium Carbonate

CaCO_3 with 2.7 g/cm^3 density and 20μ particles size was used and the typical data of CaCO_3 are shown in Appendix I.

3.1.4 Kaolin

Kaolin with 2.6 g/cm^3 density and 15μ particle size was used and the typical data of kaolin are shown in Appendix I.

3.1.5 Talc

Talc with density of 2.8 g/cm^3 and 10μ particle size was used and the typical data of talc are shown in Appendix I.

3.1.6 Barium Sulfate

BaSO_4 with density of 4.3 g/cm^3 and 12μ particle size was used and the typical data of BaSO_4 are shown in Appendix I.

3.2 Apparatus and Equipment

1. Two-roll mill, LAB TECH
2. Tensile tester, LLOYD Model LS 500
3. Elongation tester, LLOYD Model LS 500
4. Hardness tester, DUROTRONIC Model 2000
5. Scanning electron microscopy, JOEL Model JSM-5300

3.3 Experimental

Various composites were prepared by varying the amounts of EVA, LDPE and fillers such as CaCO_3 , kaolin, talc and BaSO_4 (Table 3.1). All components of the preparations were in part by weight of total amount of EVA, LDPE and fillers. Finally, mechanical properties such as tensile strength in machine direction, tensile strength in transverse direction, ultimate elongation in machine direction, ultimate elongation in transverse direction and hardness (Shore A) of each composition were measured and compared with those from commercial resin.

Table3.1 Composition by weight of prepared composites

Sample No.	EVA		LDPE	FILLERS			
	N8038	N8045	JJ4324	CaCO ₃	Kaolin	Talc	BaSO ₄
1	30	-	5	65	-	-	-
2	25	-	5	70	-	-	-
3	20	-	5	75	-	-	-
4	30	-	5	-	65	-	-
5	25	-	5	-	70	-	-
6	20	-	5	-	75	-	-
7	30	-	5	-	-	65	-
8	25	-	5	-	-	70	-
9	20	-	5	-	-	75	-
10	30	-	5	-	-	-	65
11	25	-	5	-	-	-	70
12	20	-	5	-	-	-	75
13	-	30	5	65	-	-	-
14	-	25	5	70	-	-	-
15	-	20	5	75	-	-	-
16	-	30	5	-	65	-	-
17	-	25	5	-	70	-	-
18	-	20	5	-	75	-	-
19	-	30	5	-	-	65	-
20	-	25	5	-	-	70	-
21	-	20	5	-	-	75	-
22	-	30	5	-	-	-	65
23	-	25	5	-	-	-	70
24	-	20	5	-	-	-	75

3.4 Preparation of Polymer Composites

The polymer composites were prepared by blending all of the components using a two-roll mill at 130°C [30]. The mixture of EVA/LDPE was first subjected to preheat for 1-2 minutes and then fillers were added. The mixing process was carried out for at least 20 minutes. Distance between the rolls (the roll nip) was adjusted to facilitate mixing. The two-roll mill was set at 130°C, 15 inches width of rolls and distance between the rolls at 2 millimeters.

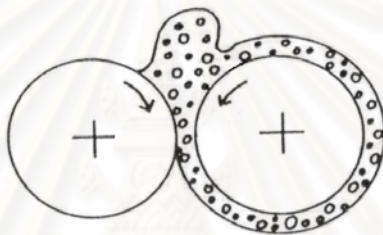


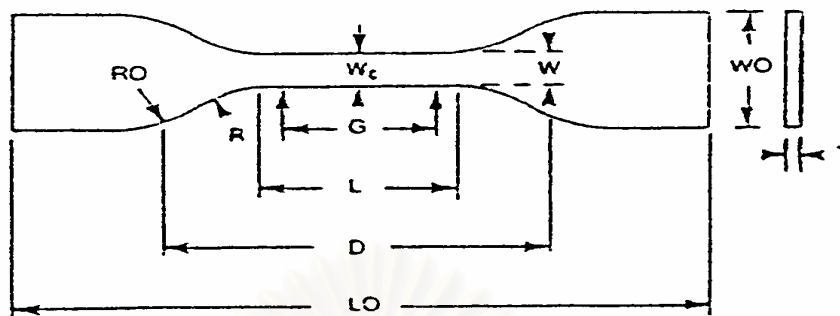
Figure 3.1 Two-roll mill

3.5 Mechanical Testing

Mechanical properties of the composites were measured by the following ASTM test methods.

3.5.1 Tensile Testing

Tensile strength of the sample was determined as per ASTM D638-97, at 25°C and 60% humidity using dumbbell-shaped test pieces, Type IV, as illustrated in Figure 3.1. The testing was performed on a tensile testing machine with a crosshead speed of testing, $50 \pm 10\%$ mm/min [31-32].



W	-Width of narrow section	6 mm.
L	-Length of narrow section	33 mm.
W _o	-Width of overall min	19 mm.
L _o	-Length of overall min	115 mm.
G	-Gage length	24 mm.
D	-Distance between grips	64 mm.
R	-Radius of fillet	14 mm.
R _o	-Outer radius	25 mm.
T	-Thickness	2 mm.

Figure 3.2 Schematic dimension of tensile test specimen

Both of tensile strength and ultimate elongation testing are analyzed on composite sheet in 2 line, machine direction (MD) and transverse direction (TD).

3.5.2 Ultimate Elongation Testing

Specimen for ultimate elongation test was determined as per tensile strength specimen (ASTM D638-97) [32-33].

3.5.3 Hardness Testing

Hardness of the sample was determined as per ASTM D2240-97 Shore A. The test specimen shall be at least 6 mm. in thickness. The lateral dimensions of the specimen should be sufficient to permit measurements at least 12 mm. from any edge unless it is known that identical results are obtained when measurements are made at a lesser distance from an edge. The surfaces of the specimen should be flat and parallel over a sufficient area to permit the presser foot to contact the specimen [34-35].

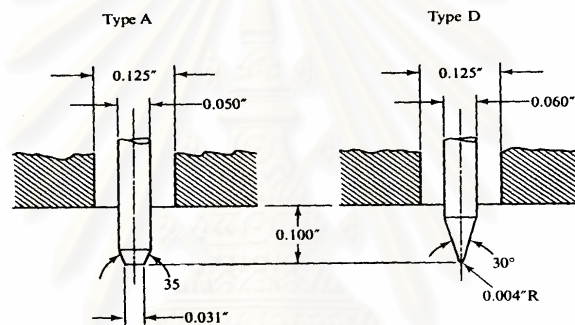


Figure 3.3 Type A and type D indentors for the Shore durometer

The specimen was placed on a hard, horizontal surface. The durometer was held in a vertical position with the point of the indenter at less than 12 mm. from any edge of the specimen. The presser foot was applied to the specimen as rapidly as possible, without shock, the foot was kept parallel to the surface of the specimen. Just sufficient pressure was applied to obtain a firm contact between the presser foot and the specimen, the scale reading is to be within 15 s. The conditions in testing were shown as follows:

Temperature	25°C
Humidity	60%

One measurement was made at each of five different points distributed over the specimen of at least 6 mm. apart using the median of these measurements for the hardness value [36].

3.6 Scanning Electron Microscopy

The scanning electron microscopy (SEM) was used to investigate the fracture surface of the composite sheet from the tensile testing. Samples for SEM were mounted on a SEM stub using a double-side tape and the fractured specimens were coated with gold. The SEM was operated at 20 kV, 1500 X

3.7 Manufacturing of the Finished Products

In automotive industry, mass back product is produced by calendering machine because mass back is width sheet. The process in the production of sheet of accurate gauge was carried out by passing the compound between rotating rolls. Often, more than one nip is required to give sheet of the required accuracy, and multi-roll machines are then used.

Calendered sheet is usually highly orientated, and exhibits anisotropic behavior in its physical properties.

CHAPTER IV

RESULTS AND DISCUSSIONS

4.1 Mechanical Properties of Ethylene Vinyl Acetate Copolymer/Low-Density Polyethylene/Filler Composites

The purpose of this research is to choose the type of filler, grade of EVA and suitable ratio of EVA/LDPE/filler for mass back use in automotive industry. Accordingly, the tensile strength, ultimate elongation and hardness are the most important properties among the others. Thus its mechanical properties have been measured and used as the benchmarks of this study. These are as follows:

Tensile strength	- MD	> 300 psi
	- TD	> 200 psi
Ultimate elongation	- MD	> 400 %
	- TD	> 350 %
Hardness		75-85 Shore A

4.2 Effect of Filler Content on Mechanical Properties of Polymer Composite

In general, polymer composite contained less than 60% of filler. It was found that tensile strength increased while elongation decreased with increasing of the filler content. Serenko [38] used filler up to 90 wt% content to study mechanical properties. The effect of filler content higher than 60 wt% on both plastic and elastic properties were deformation, tensile strength, impact

strength and flexural modulus. Therefore, this experiment uses 65-75% of filler in the polymer composite. The compositions of polymer are shown in Table 3.1 in Chapter 3.

4.2.1 Tensile Properties

Tables 4.1-4.4 and Figures 4.1-4.8 showed that the tensile strength and ultimate elongation of polymer composites decrease with increasing filler content. The EVA(N8038)/LDPE/65% CaCO₃ composites have higher TS-MD, TS-TD, EL-MD and EL-TD than EVA(N8038)/LDPE/75% CaCO₃ composites by ca 12.28%, 10.83%, 9.48% and 10.85%, respectively. The EVA(N8045)/LDPE/65% CaCO₃ composites have higher TS-MD, TS-TD, EL-MD and EL-TD than EVA(N8045)/LDPE/75% CaCO₃ composites ca 23.14%, 26.28%, 7.56% and 13.03%, respectively. It is well known that an increase in loading of filler to polymer tends to deteriorate mechanical properties such as yield and ultimate strength and fracture toughness. Maiti et al. [39] studied the mechanical properties of iPP/CaCO₃ composites and they found that tensile modulus increased while tensile yield and ultimate strength decreased with increasing CaCO₃ content. These property changes are due to three main reasons. Firstly, the effect of filler is to dilute the properties of the polymer by reducing the effective cross sectional area of the specimens and agglomerates that weaken the compound. Secondary, these agglomerates act as stress concentrators because internal stresses occur around the particles causing crack initiation that lead to rupture of the specimens. Finally the adhesion of the filler surface and polymer matrix is also important. When adhesion is poor, upon the application of a force the separation at the particle-matrix interface occurs. This

brings about voiding or debonding and finally to crack propagation in the bulk of specimens [40].

Table 4.1 Tensile strength and ultimate elongation of EVA/LDPE/CaCO₃ composites

EVA	CaCO ₃ (%)	TS-MD	TS-TD	EL-MD	EL-TD
N8038	65	469.0	429.7	473.3	453.7
	70	439.3	408.0	450.7	432.3
	75	417.7	387.7	432.3	409.3
N8045	65	307.0	302.7	493.7	459.7
	70	275.0	271.0	475.0	432.7
	75	249.3	239.7	459.0	406.7

Table 4.2 Tensile strength and ultimate elongation of EVA/LDPE/kaolin composites

EVA	Kaolin (%)	TS-MD	TS-TD	EL-MD	EL-TD
N8038	65	398.7	360.3	482.7	463.0
	70	379.3	318.3	466.0	433.3
	75	358.0	308.7	449.3	414.3
N8045	65	268.7	247.3	514.0	482.7
	70	242.0	221.7	493.7	467.7
	75	221.0	202.3	471.7	441.7

Table 4.3 Tensile strength and ultimate elongation of EVA/LDPE/talc composites

EVA	Talc (%)	TS-MD	TS-TD	EL-MD	EL-TD
N8038	65	441.3	418.3	459.0	438.3
	70	411.7	394.3	441.3	425.0
	75	388.7	371.3	425.0	391.7
N8045	65	300.3	283.7	491.7	443.7
	70	272.0	250.7	470.0	421.3
	75	241.0	227.0	453.7	402.0

Table 4.4 Tensile strength and ultimate elongation of EVA/LDPE/BaSO₄ composites

EVA	BaSO ₄ (%)	TS-MD	TS-TD	EL-MD	EL-TD
N8038	65	485.3	459.7	456.0	417.7
	70	456.7	426.3	437.0	388.0
	75	421.7	398.0	413.3	364.7
N8045	65	332.0	300.3	465.0	433.3
	70	315.0	275.0	440.3	414.0
	75	284.7	256.0	422.7	393.3

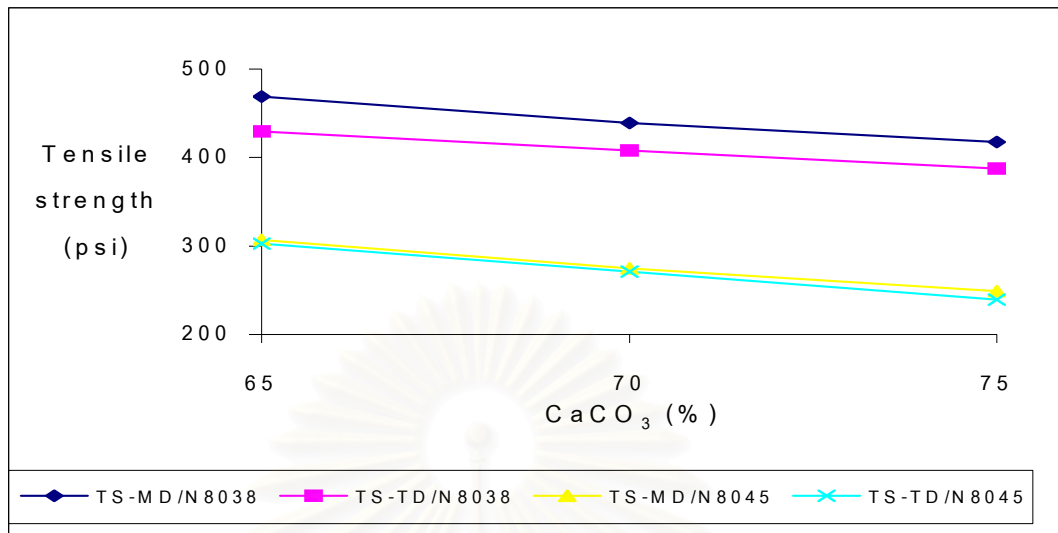


Figure 4.1 Effect of CaCO₃ on tensile strength of polymer composites

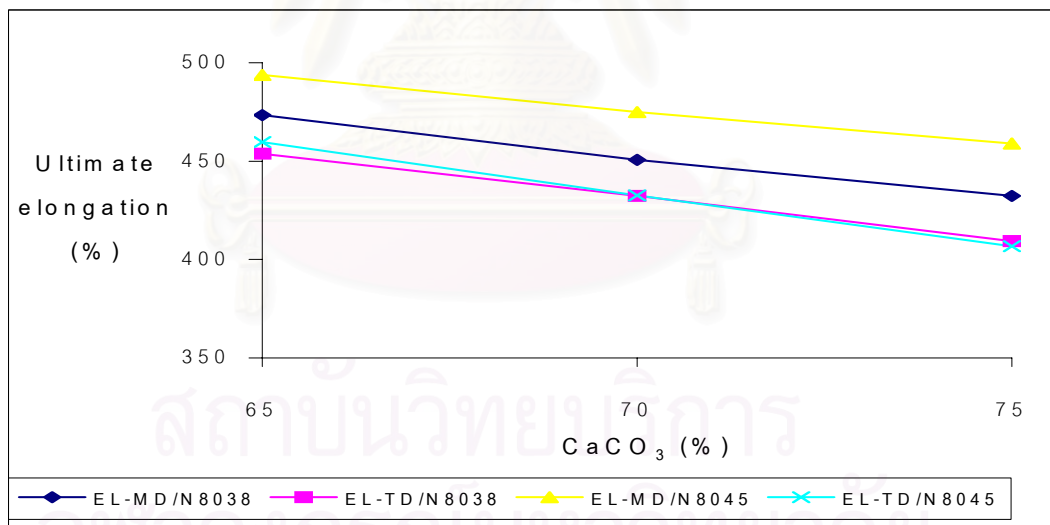


Figure 4.2 Effect of CaCO₃ on ultimate elongation of polymer composites

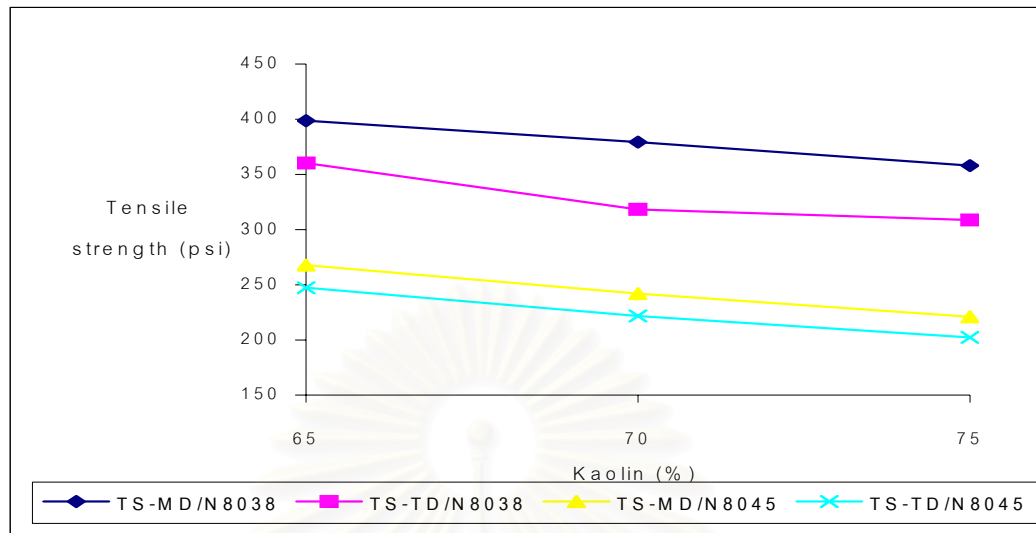


Figure 4.3 Effect of kaolin on tensile strength of polymer composites

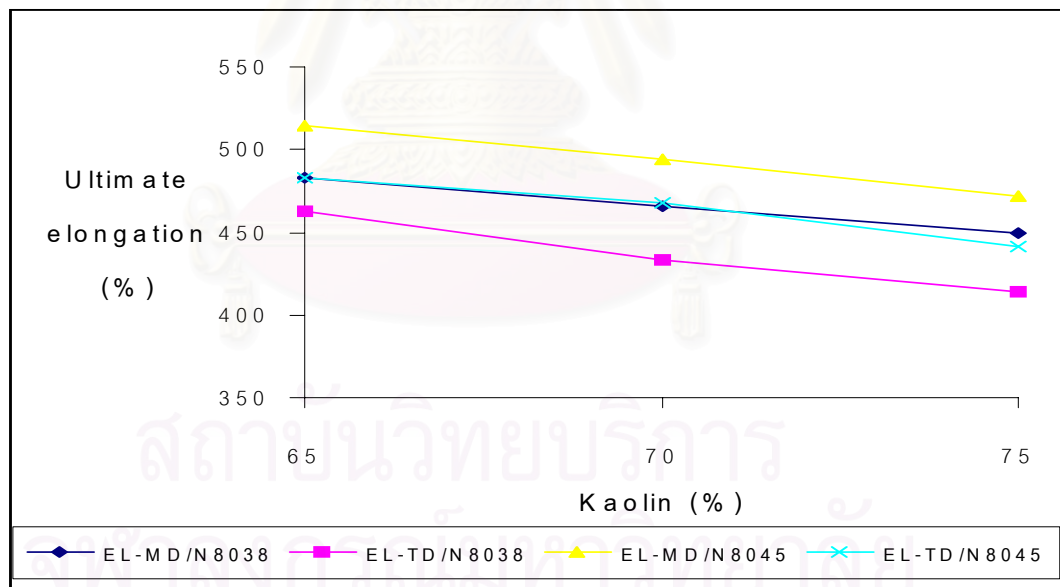


Figure 4.4 Effect of kaolin on ultimate elongation of polymer composites

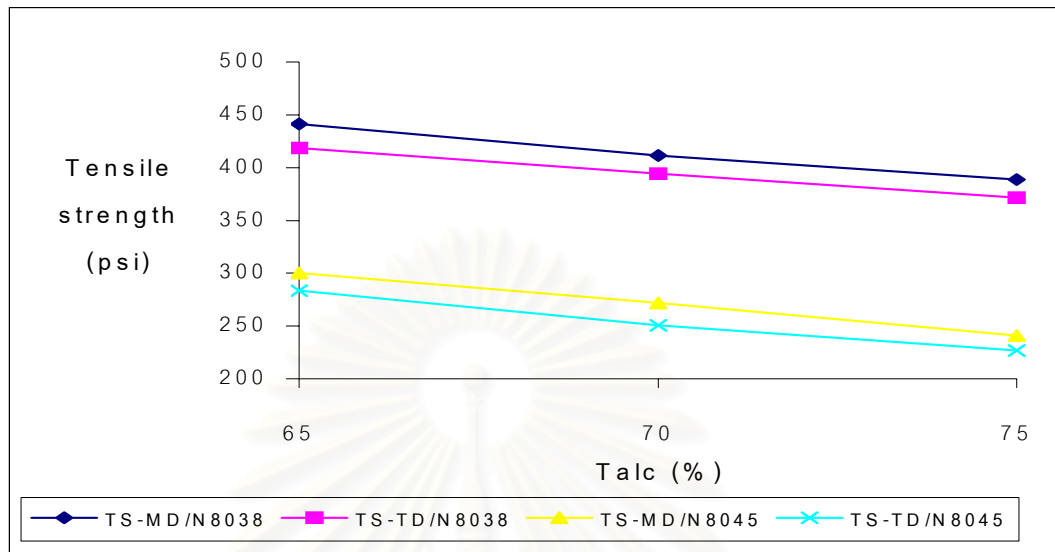


Figure 4.5 Effect of talc on tensile strength of polymer composites

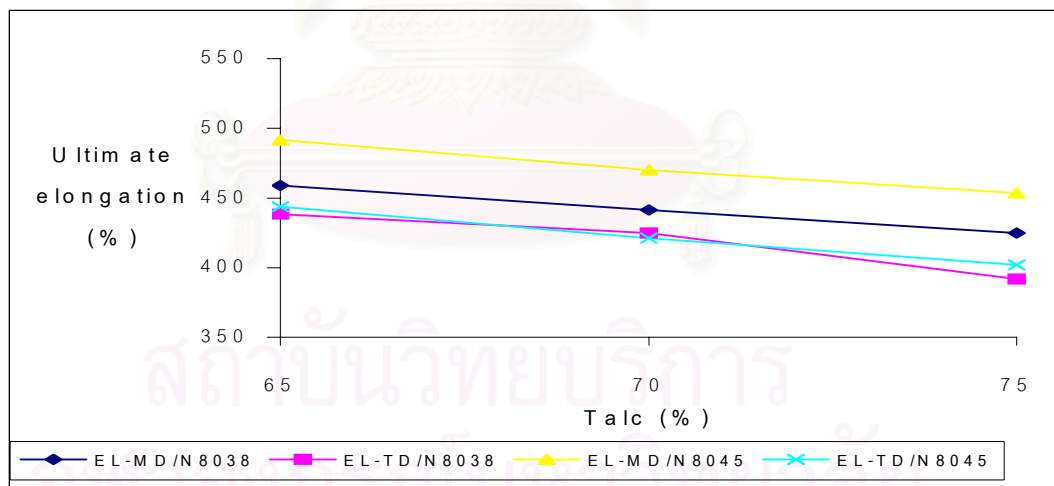


Figure 4.6 Effect of talc on ultimate elongation of polymer composites

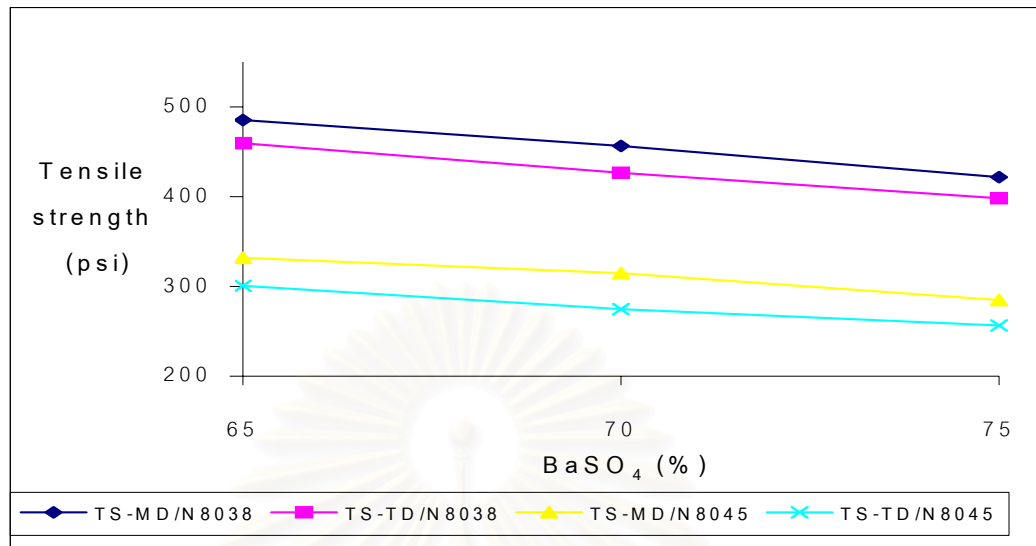


Figure 4.7 Effect of BaSO₄ on tensile strength of polymer composites

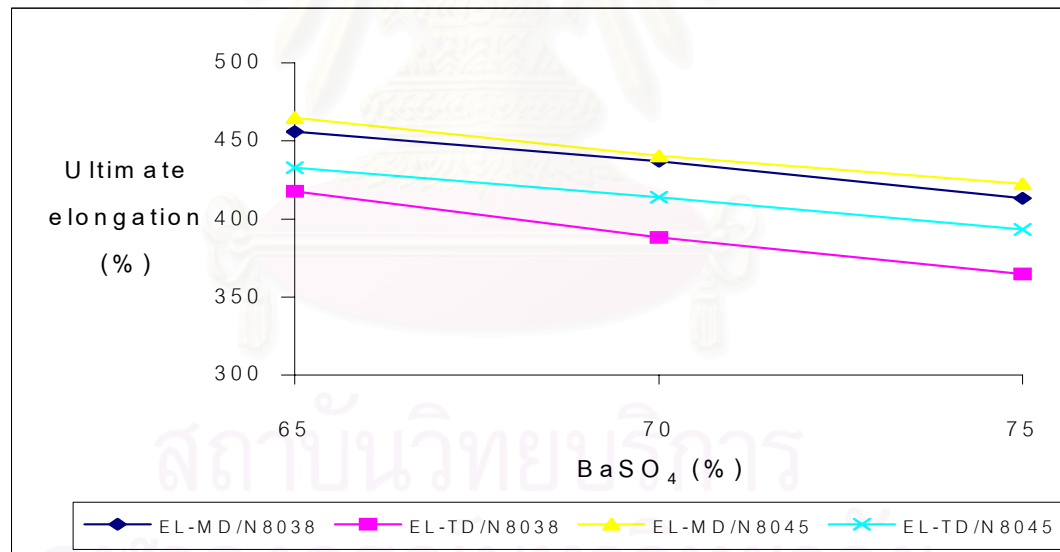


Figure 4.8 Effect of BaSO₄ on ultimate elongation of polymer composites

4.2.2 Hardness Properties

Tables 4.5-4.8 and Figures 4.9-4.12 present the hardness properties as a function of each filler content. The concentration of filler was varied from the 65, 70 and 75%. The hardness properties tend to increase with increasing the filler content. The EVA(N8038)/LDPE/75% talc composite has higher hardness than EVA(N8038)/LDPE/65% talc by ca 4.08% and EVA(N8045)/LDPE/75% talc composite has higher than EVA(N8045)/LDPE/65% talc composite by ca 4.46%. Due to surface of filler is harder than that of polymer, therefore when filler is mixed with polymer, the surface of composite is stronger than that of the polymer alone [41].

Table 4.5 Hardness of EVA/LDPE/CaCO₃

EVA	CaCO ₃ (%)	HD
N8038	65	86.7
	70	89.0
	75	91.7
N8045	65	85.3
	70	87.3
	75	90.0

Table 4.6 Hardness of EVA/LDPE/kaolin

EVA	Kaolin (%)	HD
N8038	65	84.7
	70	86.3
	75	88.7
N8045	65	81.7
	70	83.0
	75	85.7

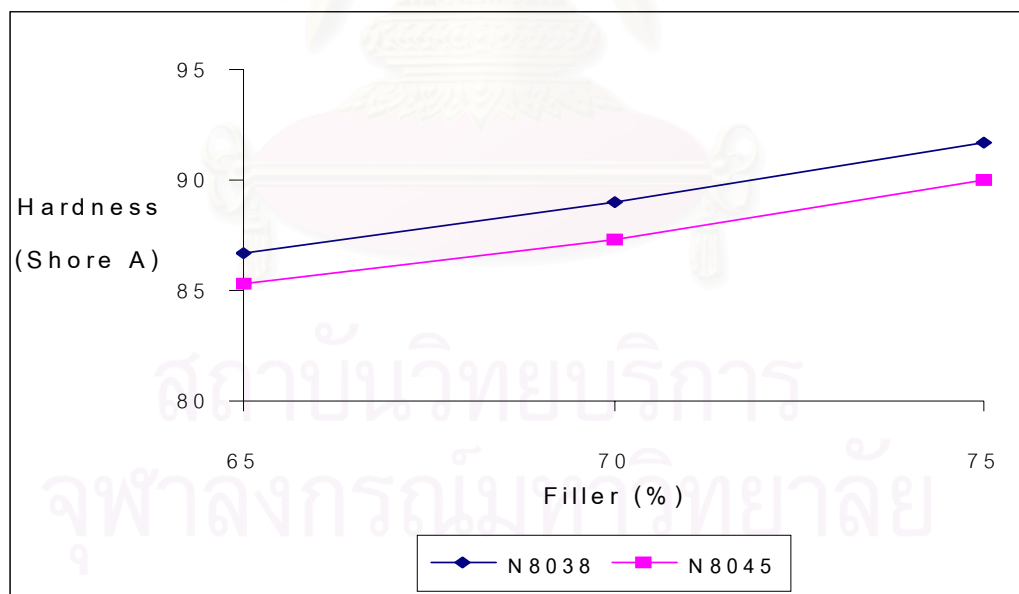
Table 4.7 Hardness of EVA/LDPE/talc

EVA	Talc (%)	HD
N8038	65	83.3
	70	84.3
	75	86.7
N8045	65	80.7
	70	82.3
	75	84.3

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Table 4.8 Hardness of EVA/LDPE/BaSO₄

EVA	BaSO ₄ (%)	HD
N8038	65	86.3
	70	87.3
	75	90.3
N8045	65	83.3
	70	85.3
	75	87.3

**Figure 4.9** Effect of CaCO₃ on hardness of polymer composites

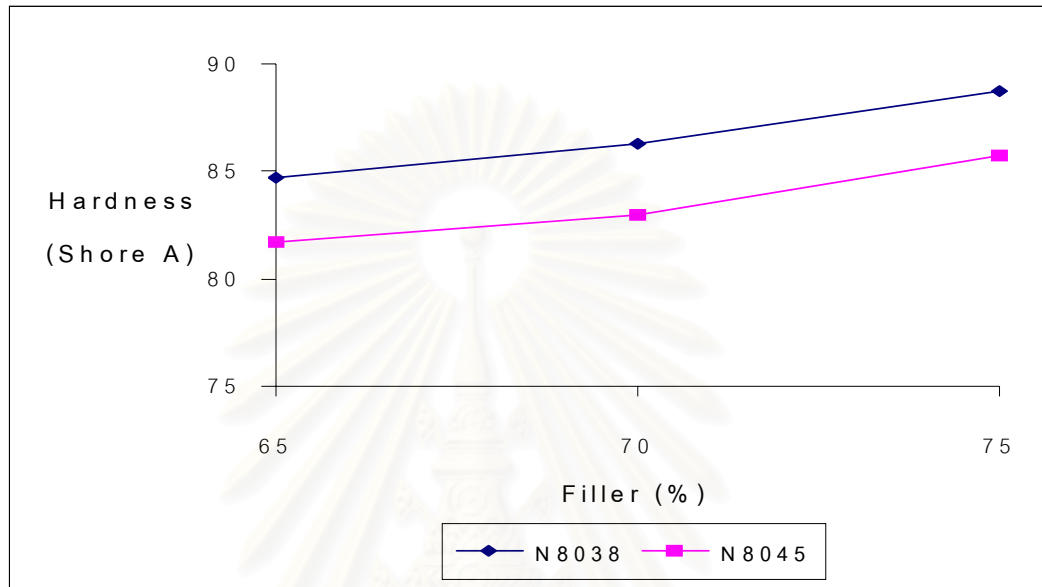


Figure 4.10 Effect of kaolin on hardness of polymer composites

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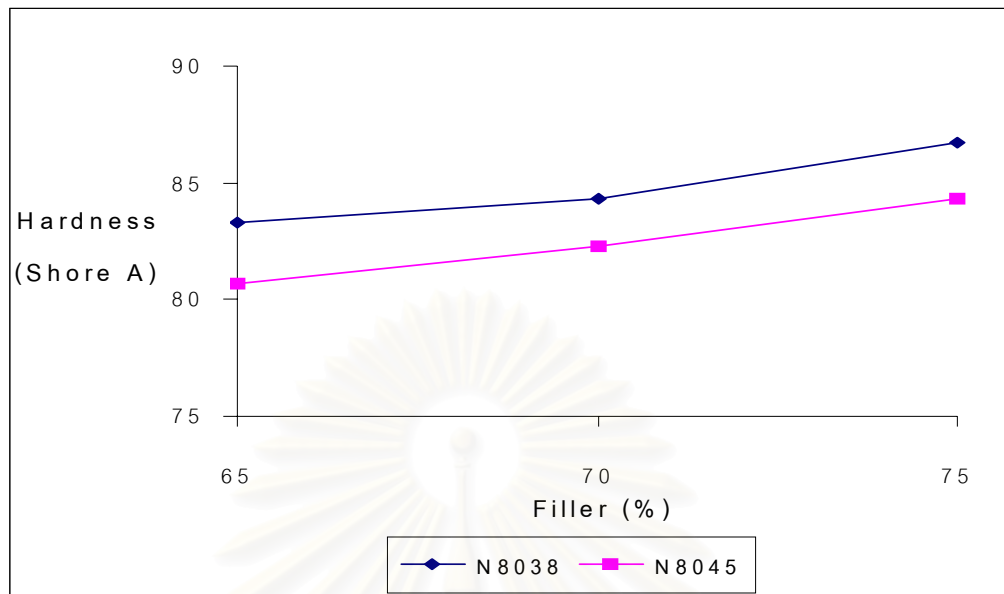


Figure 4.11 Effect of talc on hardness of polymer composites

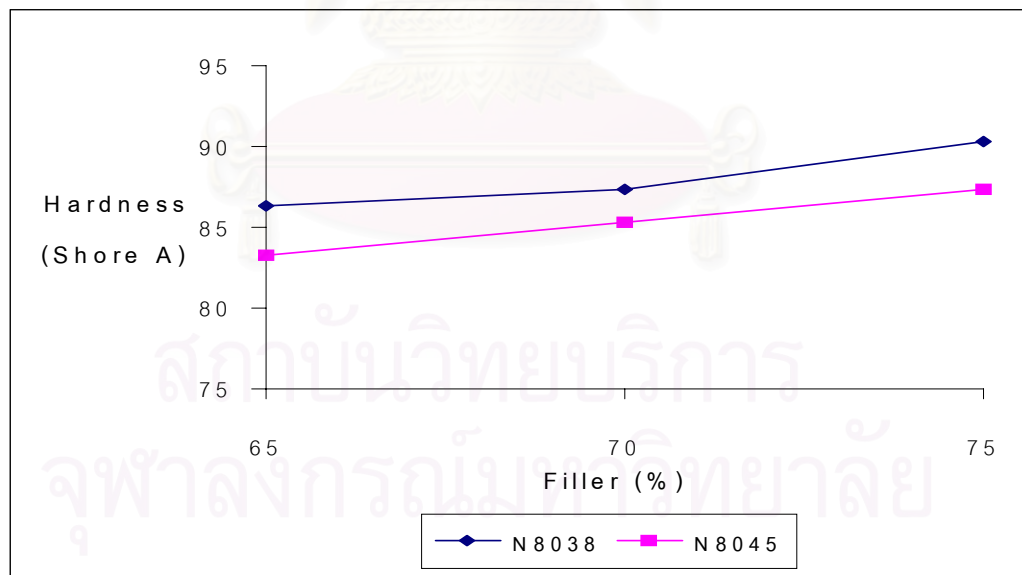


Figure 4.12 Effect of BaSO₄ on hardness of polymer composites

4.3 Effect of Filler Type on Mechanical Properties of Polymer Composite

4.3.1 Tensile Strength

Filler particle shape, particle size and interfacial bonding affect tensile strength of filled plastic compositions. At equivalent volume loading, small filler particle sizes give better tensile strength than larger particle size due to the surface area of filler increased as decreasing particle size. Void occurred after dewetting. It found that the particle size increased while void space structure increased with extra decreasing in its component material. Higher filler surface areas have stronger filler-to-matrix bonding also result in higher tensile strength composition [42]. The following fillers are listed in the order of their increasing particle size (from Tables 4-7 in Appendix I); CaCO_3 >kaolin> BaSO_4 >talc. Moreover, shape of CaCO_3 and BaSO_4 are sphere while talc and kaolin are lamellae. Fundamentally, tensile strength has been affected by its particle size which higher tensile strength can be obtained from sphere more than lamellae filler. From the experiment, Tables 4.9-4.10 and Figures 4.13-4.16 show the TS-MD and TS-TD of EVA(N8038)/LDPE/65% kaolin composite was 398.7 and 360.3 psi, EVA(N8038)/LDPE/65% talc composite was 441.3 and 418.3 psi, EVA(N8038)/LDPE/65% CaCO_3 composite was 469.0 and 429.7 psi and EVA(N8038)/LDPE/65% BaSO_4 composite was 485.3 and 459.7 psi. It was note that the TS-MD and TS-TD are the highest for the BaSO_4 -filled composite and the lowest for the kaolin-filled composite. Similar results are obtained at 70 and 75% filler content.

Table 4.9 Tensile strength of polymer composite with EVA as N8038

	Sample No.	TS-MD	TS-TD
N8038 30%	1	469.0	429.7
	4	398.7	360.3
	7	441.3	418.3
	10	485.3	459.7
N8038 25%	2	439.3	408.0
	5	379.3	318.3
	8	411.7	394.3
	11	456.7	426.3
N8038 20%	3	417.7	387.7
	6	358.0	308.7
	9	388.7	371.3
	12	421.7	398.0

Table 4.10 Tensile strength of polymer composite with EVA as N8045

	Sample No.	TS-MD	TS-TD
N8045 30%	13	307.0	302.7
	16	268.7	247.3
	19	300.3	283.7
	22	332.0	300.3
N8045 25%	14	275.0	271.0
	17	242.0	221.7
	20	272.0	250.7
	23	315.0	275.0
N8045 20%	15	249.3	239.7
	18	221.0	202.3
	21	241.0	227.0
	24	284.7	256.6

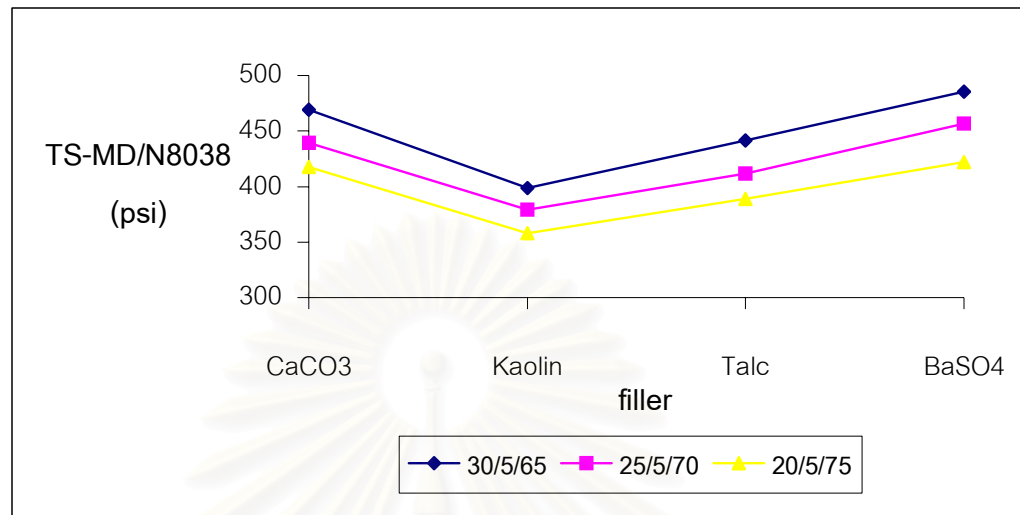


Figure 4.13 Effect of filler type on tensile strength-MD with EVA as N8038

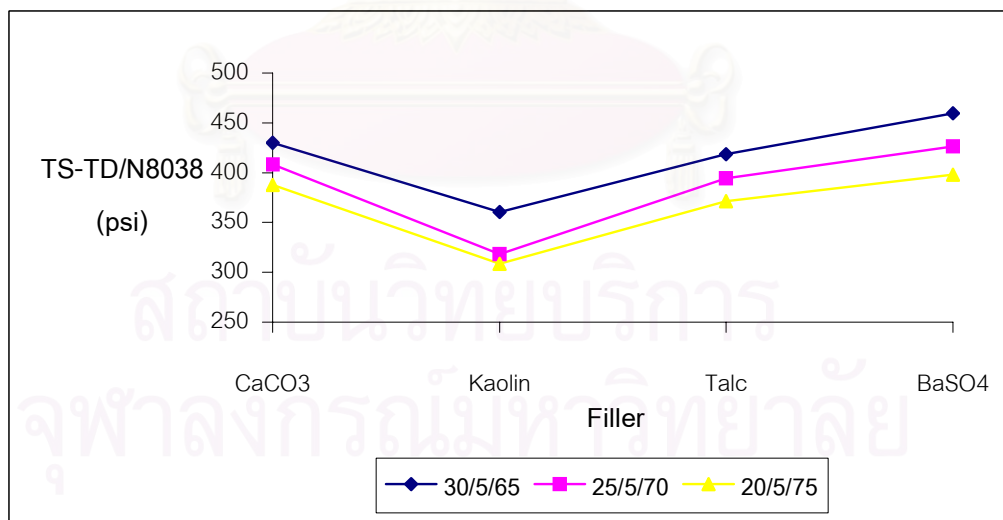


Figure 4.14 Effect of filler type on tensile strength-TD with EVA as N8038

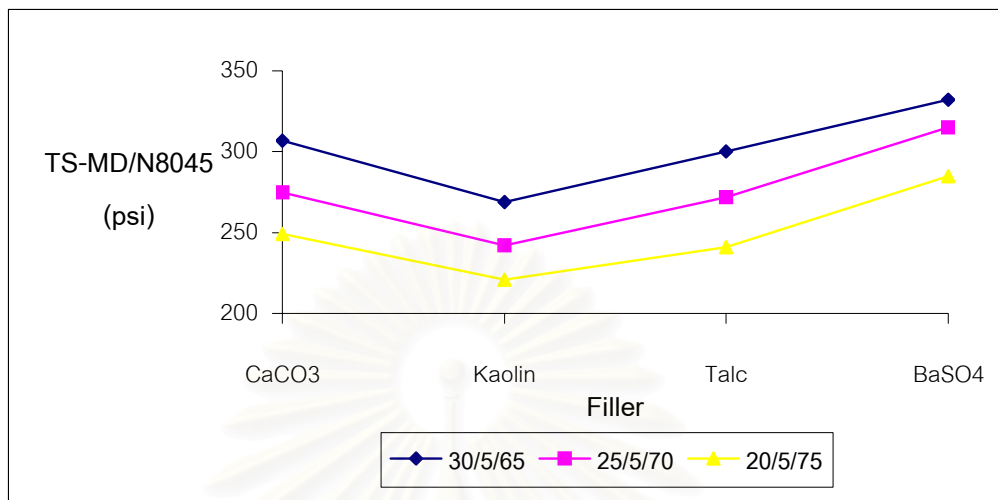


Figure 4.15 Effect of filler type on tensile strength-MD with EVA as N8045

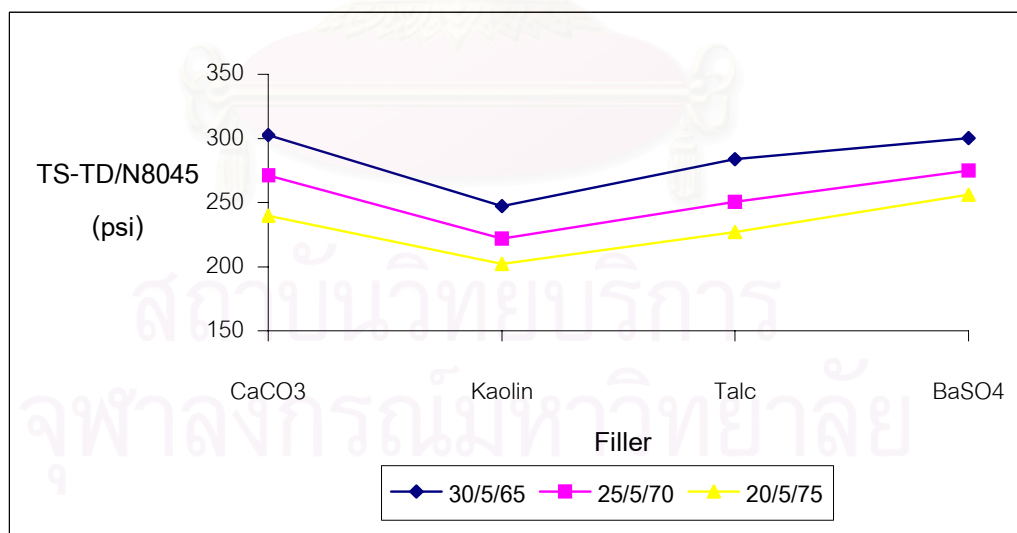


Figure 4.16 Effect of filler type on tensile strength-TD with EVA as N8045

4.3.2 Ultimate Elongation

The small particle size of filler was easier to agglomerate than the large one. Thus, agglomeration of small particles deteriorated mechanical properties [42]. Tables 4.11-4.12 and Figures 4.17-4.20 showed the effect of particle size and shape of filler on ultimate elongation of polymer composite. Kaolin and talc have flake shape at 15 and 10 μ particle size while CaCO_3 and BaSO_4 have sphere shape at 20 and 12 μ particle size. Small particles of filler affect the ultimate elongation lower than the large one when shape of fillers are the same. The elongation in machine direction of 65% kaolin, talc, CaCO_3 and BaSO_4 with the compound of EVA grade N8038 and LDPE were 514.0, 491.7, 493.7 and 465.0% respectively.

Table 4.11 Ultimate elongation of polymer composite with EVA as N8038

	Sample No.	EL-MD	EL-TD
N8038 30%	1	473.3	453.7
	4	482.7	463.0
	7	459.0	438.3
	10	456.0	417.7
N8038 25%	2	450.7	432.3
	5	466.0	433.3
	8	441.3	425.0
	11	437.0	388.0
N8038 20%	3	432.3	409.3
	6	449.3	414.3
	9	425.0	391.7
	12	413.3	364.7

Table 4.12 Ultimate elongation of polymer composite with EVA as N8045

	Sample No.	EL-MD	EL-TD
N8045 30%	1	493.7	459.7
	4	514.0	482.7
	7	491.7	443.7
	10	465.0	433.0
N8045 25%	2	475.0	432.7
	5	493.7	467.7
	8	470.0	421.3
	11	440.3	414.0
N8045 20%	3	459.0	406.7
	6	471.7	441.7
	9	453.7	402.0
	12	422.7	393.3

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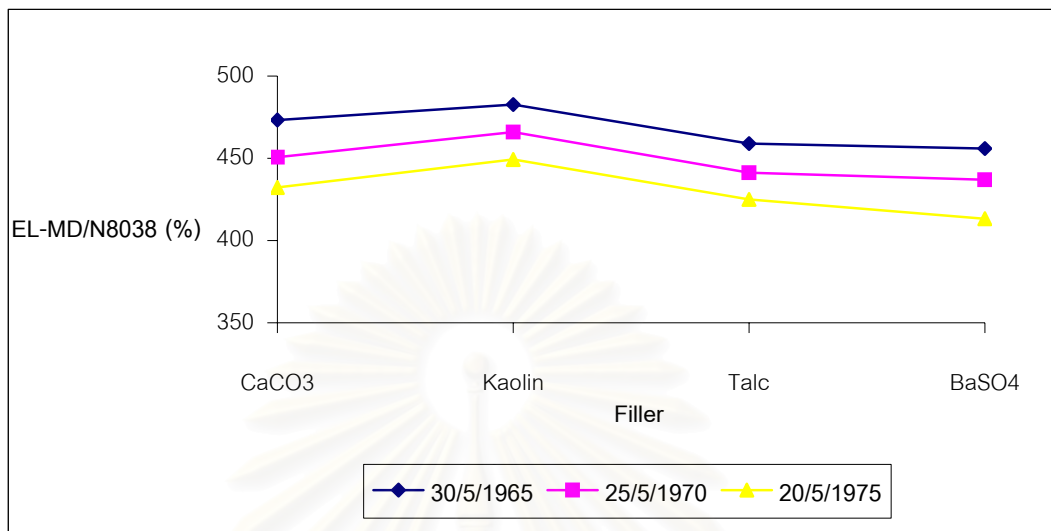


Figure 4.17 Effect of filler type on ultimate elongation-MD with EVA as N8038

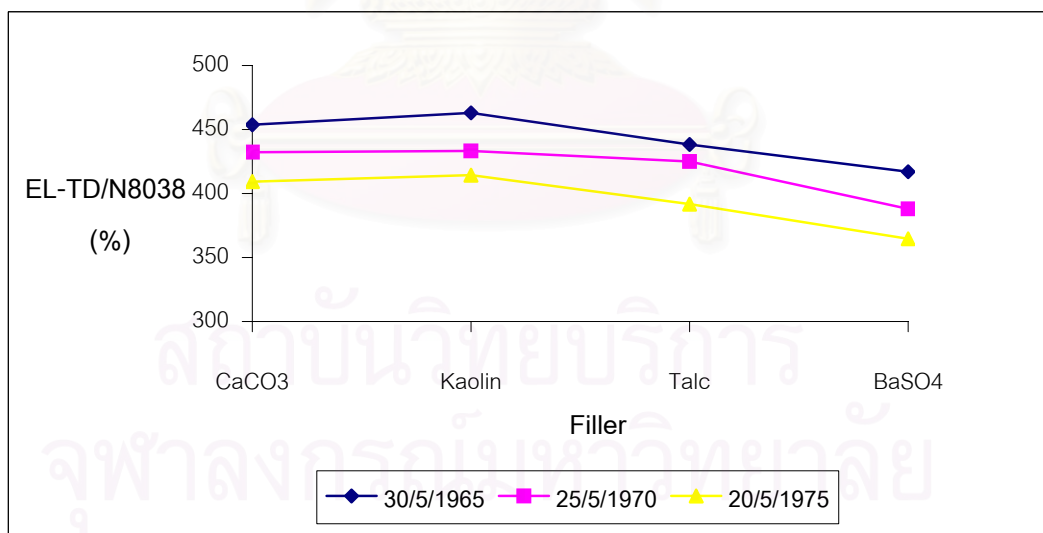


Figure 4.18 Effect of filler type on ultimate elongation-TD with EVA as N8038

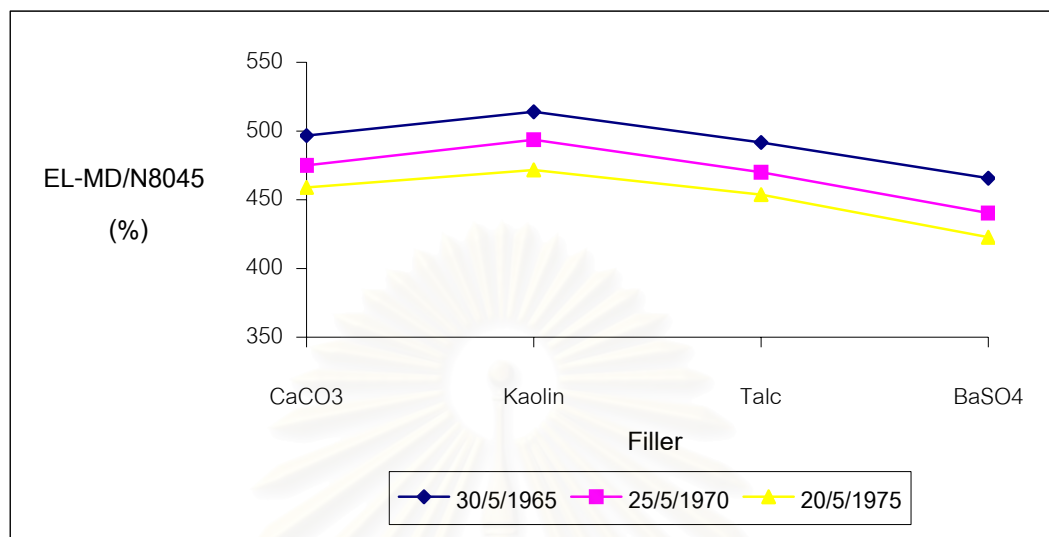


Figure 4.19 Effect of filler type on ultimate elongation-MD with EVA as N8045

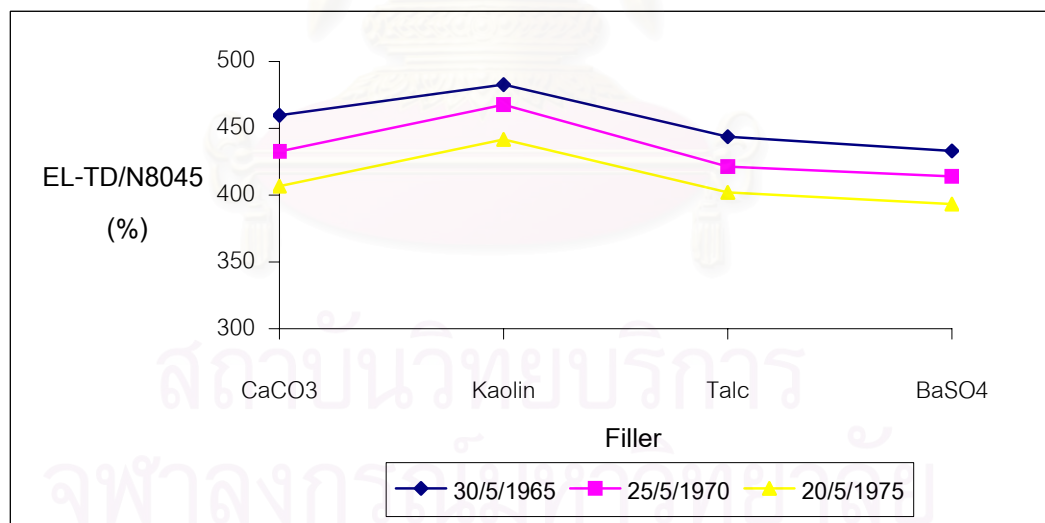


Figure 4.20 Effect of filler type on ultimate elongation-TD with EVA as N8045

4.3.3 Hardness

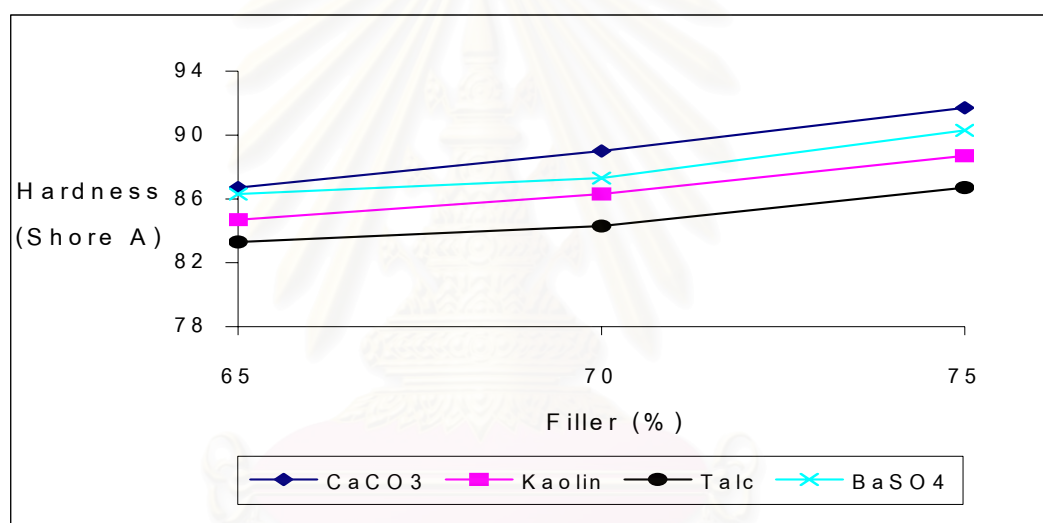
Talc is the softest known mineral for filler and it has a slippery feel. On the Mohs hardness scale, talc is the standard for a hardness of 1. Kaolin with Mohs hardness 2 is extremely soft and nonabrasive because of their platy character and the absence of massive particle with defined shape edges. BaSO_4 is a moderately soft crystalline mineral with Mohs hardness 2.5. Mohs hardness of CaCO_3 is 3 [11]. Therefore, the hardness of fillers increased in the order: talc < kaolin < BaSO_4 < CaCO_3 . Tables 4.13-4.14 and Figures 4.21-4.22 illustrate the hardness Shore A of polymer composites having different fillers. Talc gives the lowest hardness while CaCO_3 gives the highest hardness. From Table 4.13, the polymer composite composed of 30% EVA(N8038), 5% LDPE and 65% talc shows hardness Shore A of 83.3 and 84.7, 86.3, 86.7 for composite with kaolin, BaSO_4 and CaCO_3 , respectively.

Table 4.13 Hardness of polymer composites with EVA as N8038

Filler (%)	CaCO_3	Kaolin	Talc	BaSO_4
65	86.7	84.7	83.3	86.3
70	89.0	86.3	84.3	87.3
75	91.7	88.7	86.7	90.3

Table 4.14 Hardness of polymer composites with EVA as N8045

Filler (%)	CaCO ₃	Kaolin	Talc	BaSO ₄
65	85.3	81.7	80.7	83.3
70	87.3	83.0	82.3	85.3
75	90.0	85.7	84.3	87.3

**Figure 4.21** Effect of EVA as N8038 on hardness of polymer composites

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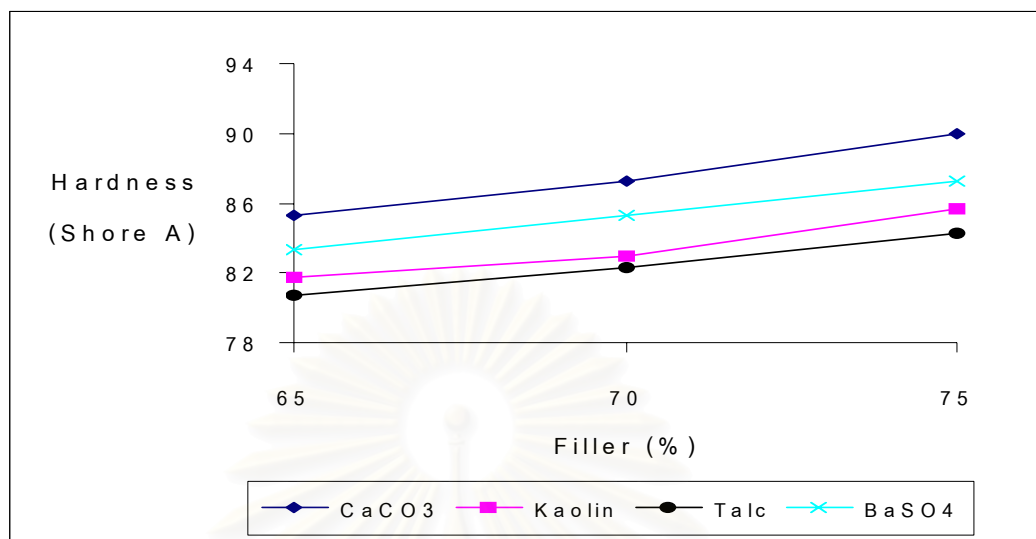


Figure 4.22 Effect of EVA as N8045 on hardness of polymer composites

4.4 Effect of Vinyl Acetate Content on Mechanical Properties of Polymer Composite

Two grades of EVA were used in this research. The N8038 has 18% VA content and the N8045 has 22% VA content. EVA has a wide range of rubberlike properties. Since the VA content controls the elasticity, it should be noted that at higher VA levels, there is extremely high elongation. This elongation controls the rubberiness and amorphous character of the copolymer. The high level of VA contents will result in the tacky and rubbery feeling of plastics. A copolymer with a very low level of VA will appear to be harder, slicker and much stiffer. The high VA content products have low tensile and modulus (stiffness) with a high degree of elongation, but little elasticity, while the opposite is true of the low VA content copolymers [43]. Tables 4.15-4.18 and Figures 4.23-4.26 showed the effect of VA content in EVA on tensile

strength and ultimate elongation. It can be seen that tensile strength decreased while ultimate elongation increased with increasing amount of VA content in the composites.

The surface hardness of EVA polymers decreased with increasing VA content. The branching and loss of crystallinity are the factors in hardness control. Tables 4.13-4.14 and Figures 4.21-4.22 show effect of VA content in EVA on hardness properties of composite. The hardness properties of polymer composites containing EVA with high VA content (N8045, 22% VA content) showed low surface hardness as a result of the acetoxy branches interference with crystallization. The EVA(N8045)/LDPE/65% talc composite has lower TS-MD, TS-TD and hardness properties than EVA(N8038)/LDPE/65% talc composite by ca 46.95%, 47.44% and 3.22% and has higher EL-MD and EL-TD by ca 7.12% and 1.23%.

Table 4.15 Mechanical properties of polymer composites with filler as CaCO_3

Filler (%)	EVA	TS-MD	TS-TD	EL-MD	EL-TD	HD
65	N8038	469.0	429.7	473.3	453.7	86.7
	N8045	307.0	302.7	493.7	459.7	85.3
70	N8038	439.3	408.0	450.7	432.3	89.0
	N8045	275.0	271.0	475.0	432.7	87.3
75	N8038	417.7	387.7	432.3	409.3	91.7
	N8045	249.3	239.7	459.0	406.7	90.0

Table 4.16 Mechanical properties of polymer composites with filler as kaolin

Filler (%)	EVA	TS-MD	TS-TD	EL-MD	EL-TD	HD
65	N8038	398.7	360.3	482.7	463.0	84.7
	N8045	268.7	247.3	514.0	482.7	81.7
70	N8038	379.3	318.3	466.0	433.3	86.3
	N8045	242.0	221.7	493.7	467.7	83.0
75	N8038	358.0	308.7	449.3	414.3	88.7
	N8045	221.0	202.3	471.7	441.7	85.7

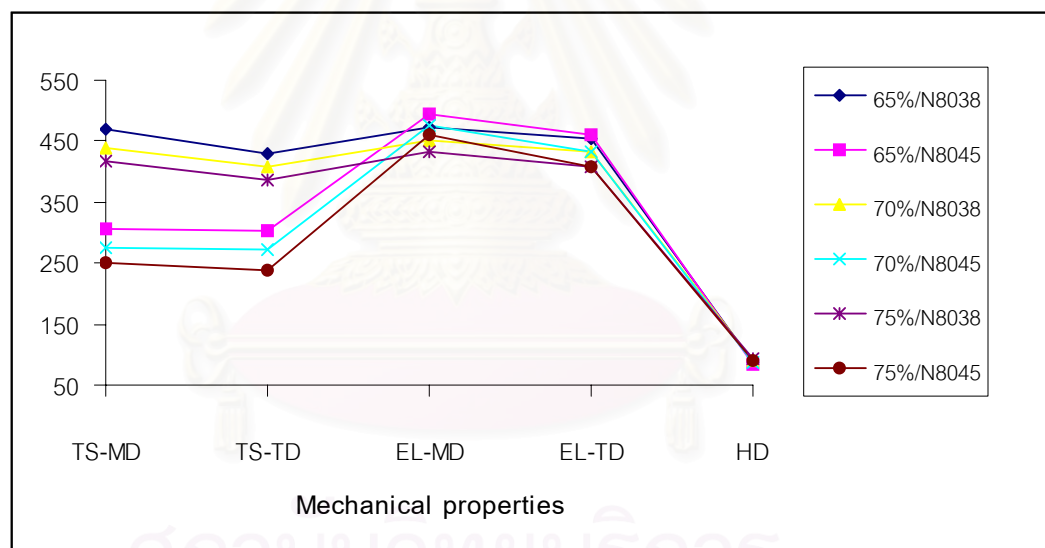
Table 4.17 Mechanical properties of polymer composites with filler as talc

Filler (%)	EVA	TS-MD	TS-TD	EL-MD	EL-TD	HD
65	N8038	441.3	418.3	459.0	438.3	83.3
	N8045	300.3	283.7	491.7	443.7	80.7
70	N8038	411.7	394.3	441.3	425.0	84.3
	N8045	272.0	250.7	470.0	421.3	82.3
75	N8038	388.7	371.3	425.0	391.7	86.7
	N8045	241.0	227.0	452.7	402.0	84.3

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Table 4.18 Mechanical properties of polymer composites with filler as BaSO₄

Filler (%)	EVA	TS-MD	TS-TD	EL-MD	EL-TD	HD
65	N8038	485.3	459.7	456.0	417.7	86.3
	N8045	332.0	300.3	465.0	433.0	83.3
70	N8038	456.7	426.3	437.0	388.0	87.3
	N8045	315.0	275.0	400.3	414.0	85.3
75	N8038	421.7	398.0	413.3	364.7	90.3
	N8045	284.77	256.0	422.7	393.3	87.3

**Figure 4.23** Effect of VA content on mechanical properties with filler as CaCO₃

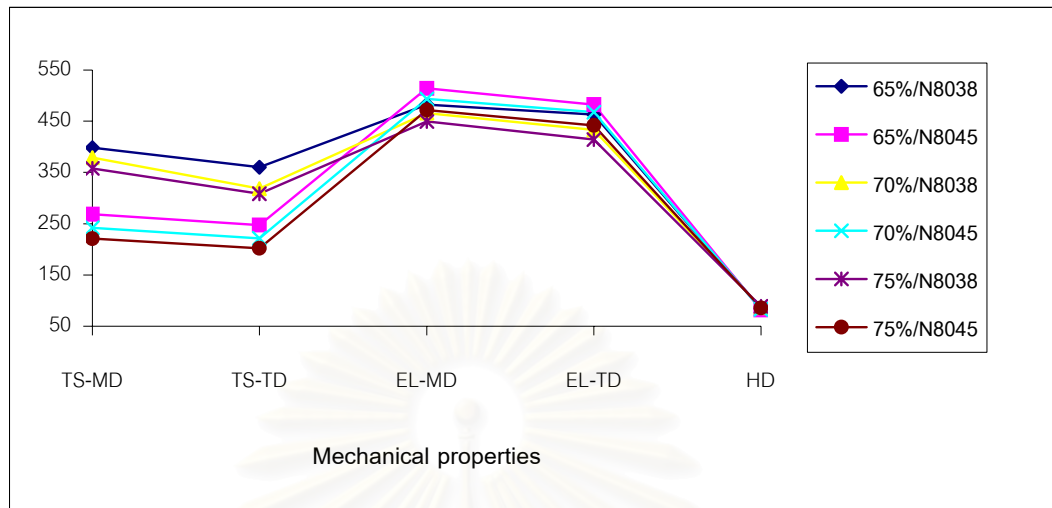


Figure 4.24 Effect of VA content on mechanical properties with filler as kaolin

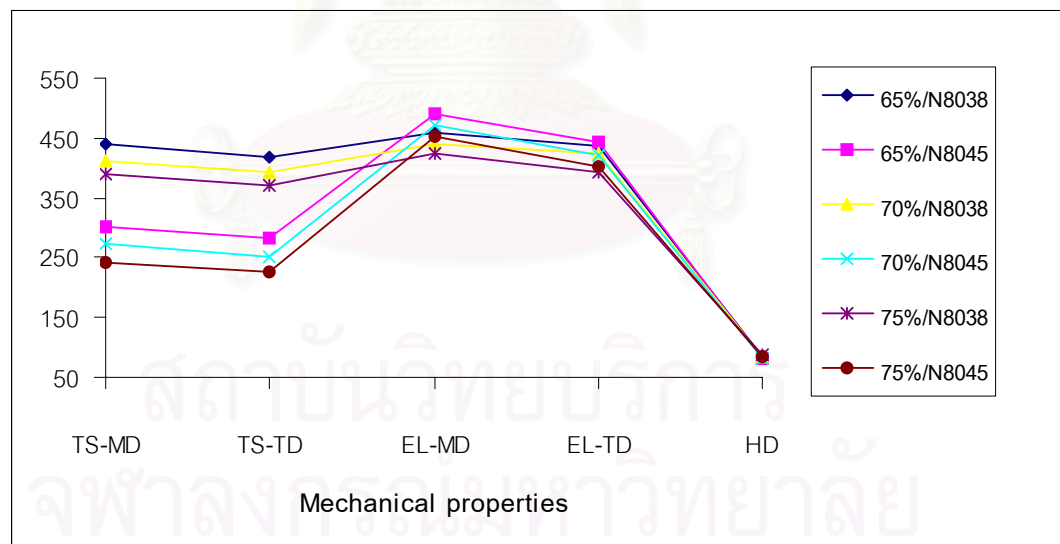


Figure 4.25 Effect of VA content on mechanical properties with filler as talc

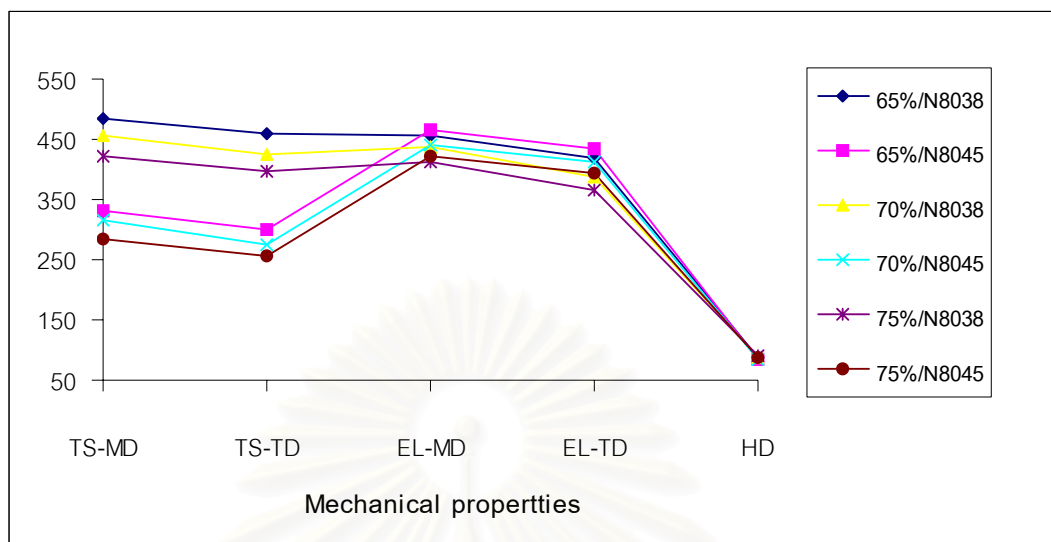


Figure 4.26 Effect of VA content on mechanical properties with filler as BaSO_4

4.5 SEM Micrographs of Polymer Composite

Tensile fracture surface of specimen was studied by SEM. The SEM micrographs in Figures 1-11 in Appendix III showed the interface of polymer composite, polymer matrix, fillers and void. The SEM micrograph of commercial resin in Figures 1 showed that polymer matrix encapsulates particles of fillers but some particles are still free and distributed in void [44]. Particle size of filler in commercial resin was estimated 5-10 μ compared with 10-20 μ in this thesis.

The SEM micrographs of polymer composites at 65, 70 and 75% talc were shown in Figures 4-6. They indicated that the higher of filler content was, the more un-encapsulated particles in polymer matrix would be. This is the reason why tensile strength and ultimate elongation of polymer composite

decreased while hardness increased. Figures 2, 3, 4 and 7 showed SEM micrographs of polymer composites with 65% of CaCO_3 , kaolin, BaSO_4 and talc. They revealed that different type of fillers had different particle size. They were in the order of $\text{CaCO}_3 > \text{kaolin} > \text{BaSO}_4 > \text{talc}$, respectively.

4.6 Comparison of Mechanical Properties with Commercial Resin

4.6.1 Mechanical Properties

From the results of this study, there were 5 optimum ratios of polymer composites containing, N8038/N8045/JJ4324/ CaCO_3 /kaolin/talc/ BaSO_4 ; 30/-/5/-/65/-/-, 30/-/5/-/-/65/-, 25/-/5/-/-/70/-, -/30/5/-/-/65/- and -/30/5/-/-/-/65. For -/30/5/-/-/65/-, this composite had lower TS-MD and was close to the lowest of specification of commercial resin, while those of 30/-/5/-/65/-/- and 25/-/5/-/-/65 ratios had hardness close to the highest of specification for commercial resin.

Table 4.19 Comparison of mechanical properties of commercial resin and prepared polymer composites

Mechanical Properties	Unit	Commercial Resin	30/-/5/-/65/-/-	30/-/5/-/-/65/-	25/-/5/-/-/70/-	-/30/5/-/-/65/-	-/30/5/-/-/-/65
TS-MD	psi	> 300	398.7	441.3	411.7	300.3	332.0
TS-TD	psi	> 200	360.3	418.3	394.3	283.7	300.3
EL-MD	%	> 400	482.7	459.0	441.3	491.7	465.0
EL-TD	%	> 350	463.0	438.3	425.0	443.7	433.0
HD	Shore A	75-85	84.7	83.3	84.3	80.7	83.3

From Table 4.19, the polymer composites, which gave suitable overall performance are 30/-/5/-/65/- and -/30/5/-/65/. In Appendix I, density of talc is 2.8 g/cm^3 and density of BaSO_4 is 4.3 g/cm^3 . In the processing of composite sheet at 2 mm. thickness which requires higher amount of BaSO_4 than talc, and the density of composite can be calculated as follows.

polymer composite		volume		density		
30/-/5/-/65/-	N8038	0.30	x	0.941	=	0.282
	LDPE	0.05	x	0.921	=	0.046
	Talc	0.65	x	2.8	=	<u>1.820</u>
						<u>2.148</u> g/cm^3
-/30/5/-/65	N8045	0.30	x	0.947	=	0.284
	LDPE	0.05	x	0.921	=	0.046
	BaSO_4	0.65	x	4.3	=	<u>2.795</u>
						<u>3.125</u> g/cm^3

Since automotive industry requires less weight on the vehicle as much as possible, therefore the 30/-/5/-/65/- composite gave lower density with 0.977 g/cm^3 which resulted in the decrease of weight by 68.7% when compared to that of the -/30/5/-/65 composite. Therefore, this ratio gave the highest saving result.

4.6.2 Economics Consideration

In this work, the costs of these composites are based on the costs of EVA, LDPE, and fillers in September 2000. The costs of EVA grade N8038,

LDPE grade JJ4324 and talc was 40, 48 and 25 Baht/kg, respectively. Table 4.20 indicated the cost of the composite with good mechanical properties as mentioned above. It can be seen that the cost of composites per kilogram is relatively low. In 2000, the costs of import commercial resin are 200 Baht/kg or higher depended on exchange rate. Therefore, the price of EVA/LDPE/filler composite obtained from this work will be attractive as an alternative to substitute the high price of the import resin. This work may lead to the development of other low-price composite resins in the future.

Table 4.20 Cost analysis of polymer composite obtained from this study

N8038/JJ4324/talc	Material cost in 2000 (Baht/kg)			Cost of composite (Baht/kg)
	N8038	JJ4324	Talc	
30/5/65	12.00	1.75	16.25	30.00

CHAPTER V

CONCLUSIONS AND SUGGESTIONS

5.1 Conclusions

The purpose of this research is to study the blend of EVA, LDPE and filler to obtain polymer composite, which can be used for floor mat application. From the study, each material grade is varied as follows; EVA (N8038 and N8045), LDPE (JJ4324) and filler (CaCO_3 , kaolin, talc and BaSO_4).

The ratio of EVA/LDPE/filler was varied as 30/5/65, 25/5/70 and 20/5/75, respectively. The blended material was milled by using two-roll mill machine, at temperature 130 °C, 2 mm. thickness of mass back. Mechanical properties such as tensile strength, ultimate elongation and hardness Shore A were used to evaluate its performance to find out the optimal ratio of EVA/LDPE/filler and to find out which EVA grade and which kind of filler are the most suitable ones. The conclusion could be drawn as the following.

1. Tensile strength and ultimate elongation decline while hardness increased, when the content of filler increases.
2. Tensile strength and hardness decline while ultimate elongation increase, when VA content increases.

This work found that the most suitable EVA was N8038, and the most suitable filler was talc, the study also found that the optimal ratio of blended EVA(N8038)/LDPE/talc was 30/5/65 which gave the mechanical properties: TS-MD, TS-TD, EL-MD, EL-TD and hardness Shore A as 441.3 psi, 418.3

psi, 459.0%, 438.8% and 83.3, respectively. The obtained polymer composite was shown to have mechanical properties agreed well with the specification of a commercial resin.

5.2 Suggestions

For the future study, the other factors should be suggested as follows.

1. The effect of particle size on its mechanical properties should be evaluated.
2. In order to obtain better mechanical properties, the blending of EVA with other thermoplastic elastomer such as EPDM should be studied.



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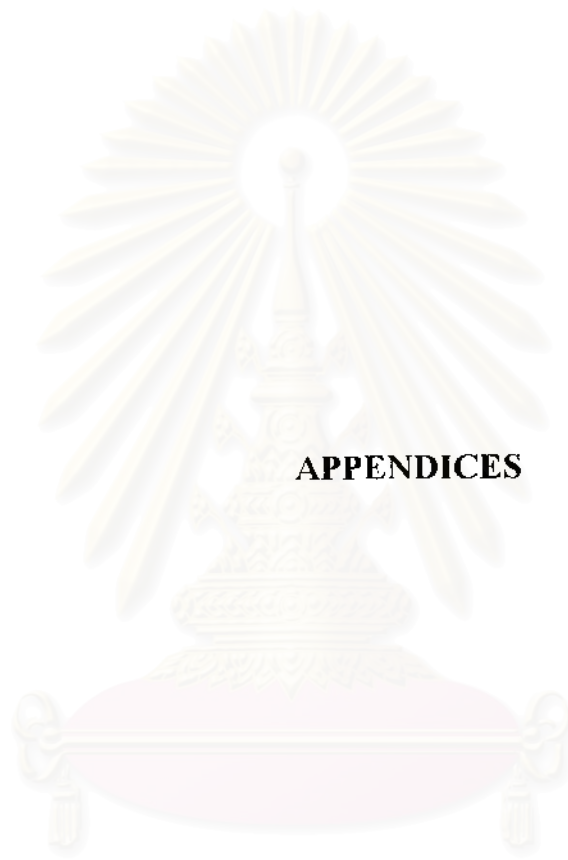
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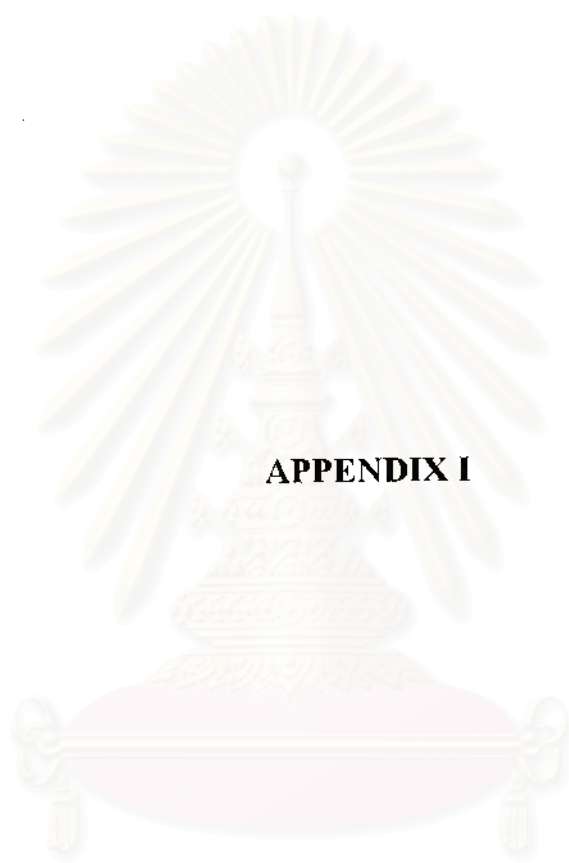
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APPENDIX I

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Table 1 Typical data of ethylene vinyl acetate grade N8038

Item	Test Method (ASTM)	Unit	Typical data
Density	D 1505	g/cm ³	0.941
Melt flow index	D 1238	g/10min	2.30
Tensile strength	D 638	psi	578
Ultimate elongation	D 638	%	700
VA content	-	% max	18
Hardness	D 2240	Shore A	78
Vicat softening temperature	D 1525	°C	58

Table 2 Typical data of ethylene vinyl acetate grade N8045

Item	Test Method (ASTM)	Unit	Typical data
Density	D 1505	g/cm ³	0.947
Melt flow index	D 1238	g/10min	2.30
Tensile strength	D 638	psi	361
Ultimate elongation	D 638	%	800
VA content	-	% max	22
Hardness	D 2240	Shore A	75
Vicat softening temperature	D 1525	°C	54

Table 3 Typical data of low-density polyethylene grade JJ4324

Item	Test Method (ASTM)	Unit	Typical data
Density	D 1505	g/cm ³	0.921
Melt flow index	D 1238	g/10min	5.50
Tensile strength	D 638	psi	1590
Ultimate elongation	D 638	%	600
Vicat softening temperature	D 1525	°C	95

Table 4 Product specification of CaCO₃

Item	Unit	Specification
Density	g/cm ³	2.7
CaCO ₃	% min	98.5
MgCO ₃	% max	0.5
SiO ₂	% max	0.2
Particle size	μ	20

Table 5 Product specification of kaolin

Item	Unit	Specification
Density	g/cm ³	2.6
SiO ₂	% min	47.4
Al ₂ O ₃	% min	38.8
Fe ₂ O ₃	% min	0.6
Loss on ignition	% max	13.2
Particle size	μ	15

Table 6 Product specification of talc

Item	Unit	Specification
Density	g/cm ³	2.8
SiO ₂	% min	42.5
Al ₂ O ₃	% min	3.0
CaO	% min	15.0
MgO	% min	23.5
Particle size	μ	10

Table 7 Product specification of BaSO₄

Item	Unit	Specification
Density	g/cm ³	4.3
Oil absorption	(g/100 g)	8
Particle size	μ	12



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APPENDIX II

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Table 8 Mechanical analysis data sheet of polymer composites

Sample No.	Analysis No.	TS-MD	TS-TD	EL-MD	EL-TD	HD
1	1	463	430	470	453	86
	2	471	427	478	451	87
	3	473	432	472	457	87
	Average	469.0	429.7	473.3	453.7	86.7
2	1	439	411	449	437	89
	2	442	408	453	428	88
	3	437	405	450	432	90
	Average	439.3	408.0	450.7	432.3	89.0
3	1	415	388	436	403	92
	2	420	390	429	410	91
	3	418	385	432	415	92
	Average	417.7	378.7	432.2	409.3	91.7
4	1	394	359	486	469	85
	2	398	362	483	459	84
	3	404	360	479	461	85
	Average	398.7	360.3	482.7	463.0	84.7
5	1	376	322	468	429	87
	2	382	318	468	435	86
	3	380	315	462	436	86
	Average	379.3	318.3	466.0	433.3	86.3
6	1	355	311	452	412	88
	2	361	306	447	414	89
	3	358	309	449	417	89
	Average	358.0	308.7	449.3	414.3	88.7

Table 8 (continued)

Sample No.	Analysis No.	TS-MD	TS-TD	EL-MD	EL-TD	HD
7	1	444	423	460	442	84
	2	439	417	462	436	83
	3	441	415	455	437	83
	Average	441.3	418.3	459.0	438.3	83.3
8	1	416	397	440	425	85
	2	411	392	443	429	84
	3	408	394	441	421	84
	Average	411.7	394.3	441.3	425.0	84.3
9	1	388	371	428	392	87
	2	386	369	421	390	87
	3	392	374	426	393	86
	Average	388.7	371.3	425.0	391.7	86.7
10	1	486	461	456	422	87
	2	483	463	459	412	86
	3	487	455	453	419	86
	Average	485.3	459.7	456.0	417.7	86.3
11	1	454	422	437	385	88
	2	457	428	435	389	87
	3	459	429	439	390	87
	Average	456.7	426.3	437.0	388.0	87.3
12	1	426	401	412	360	90
	2	520	396	418	366	90
	3	419	397	410	368	91
	Average	421.7	398.0	413.3	364.7	90.3

Table 8 (continued)

Sample No.	Analysis No.	TS-MD	TS-TD	EL-MD	EL-TD	HD
13	1	311	299	493	457	86
	2	304	303	501	463	85
	3	306	306	496	459	85
	Average	307.0	302.7	496.7	459.7	85.3
14	1	273	269	478	436	87
	2	275	274	474	432	88
	3	277	270	473	430	87
	Average	275.0	271.0	475.0	432.7	87.3
15	1	252	244	461	405	91
	2	249	236	457	407	89
	3	247	239	459	408	90
	Average	249.3	239.7	459.0	406.7	90.0
16	1	271	251	516	486	81
	2	269	247	517	483	82
	3	266	244	509	479	82
	Average	268.7	247.3	514.0	482.7	81.7
17	1	242	226	498	468	84
	2	245	219	494	470	83
	3	239	220	489	465	82
	Average	242.0	221.7	493.7	467.7	83.0
18	1	222	202	472	442	86
	2	218	206	469	440	85
	3	223	199	474	443	86
	Average	221.0	202.3	471.7	441.7	85.7

Table 8 (continued)

Sample No.	Analysis No.	TS-MD	TS-TD	EL-MD	EL-TD	/HD
19	1	306	288	496	443	80
	2	296	279	489	441	80
	3	299	284	490	447	82
	Average	300.3	283.7	491.7	443.7	80.7
20	1	268	254	472	418	83
	2	273	252	468	422	82
	3	275	246	470	424	82
	Average	272.0	250.7	470.0	421.3	82.3
21	1	244	231	455	399	85
	2	240	226	455	405	84
	3	239	224	451	402	84
	Average	241.0	227.0	453.7	402.0	84.3
22	1	328	300	467	430	84
	2	333	298	463	433	84
	3	335	303	465	436	82
	Average	332.0	300.3	465.0	433.0	83.3
23	1	319	271	443	414	85
	2	309	278	438	416	86
	3	317	276	440	412	85
	Average	315.0	275.0	440.3	414.0	85.3
24	1	286	255	426	390	88
	2	281	254	424	394	87
	3	287	259	418	396	87
	Average	284.7	256.0	422.7	393.3	87.3

Remark: TS = Tensile strength
EL = Ultimate elongation
HD = Hardness
MD = Machine direction
TD = Transverse direction



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Table 9 Mechanical properties of polymer composites with EVA as N8038

	Sample No.	TS-MD	TS-TD	EL-MD	EL-TD	HD
N8038 30%	1	469.0	429.7	473.3	453.7	86.7
	4	398.7	360.3	482.7	463.0	84.7
	7	441.3	418.3	459.0	438.3	83.3
	10	485.3	459.7	456.0	417.7	86.3
N8038 25%	2	439.3	408.0	450.7	432.3	89.0
	5	379.3	318.3	466.0	433.3	86.3
	8	411.7	394.3	441.3	425.0	84.3
	11	456.7	426.3	437.0	388.0	87.3
N8038 20%	3	417.7	387.7	432.3	409.3	91.7
	6	358.0	308.7	449.3	414.3	88.7
	9	388.7	371.3	425.0	391.7	86.7
	12	421.7	398.0	413.3	364.7	90.3

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Table 10 Mechanical properties of polymer composites with EVA as N8045

	Sample No.	TS-MD	TS-TD	EL-MD	EL-TD	HD
N8045 30%	13	307.0	302.7	493.7	459.7	85.3
	16	268.7	247.3	514.0	482.7	81.7
	19	300.3	283.7	491.7	443.7	80.7
	22	332.0	300.3	465.0	433.0	83.3
N8045 25%	14	275.0	271.0	475.0	432.7	87.3
	17	242.0	221.7	493.7	467.7	83.0
	20	272.0	250.7	470.0	421.3	82.3
	23	315.0	275.0	440.3	414.0	85.3
N8045 20%	15	249.3	239.7	459.0	406.7	90.0
	18	221.0	202.3	471.7	441.7	85.7
	21	241.0	227.0	453.7	402.0	84.3
	24	284.7	256.6	422.7	393.3	87.3

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APPENDIX III

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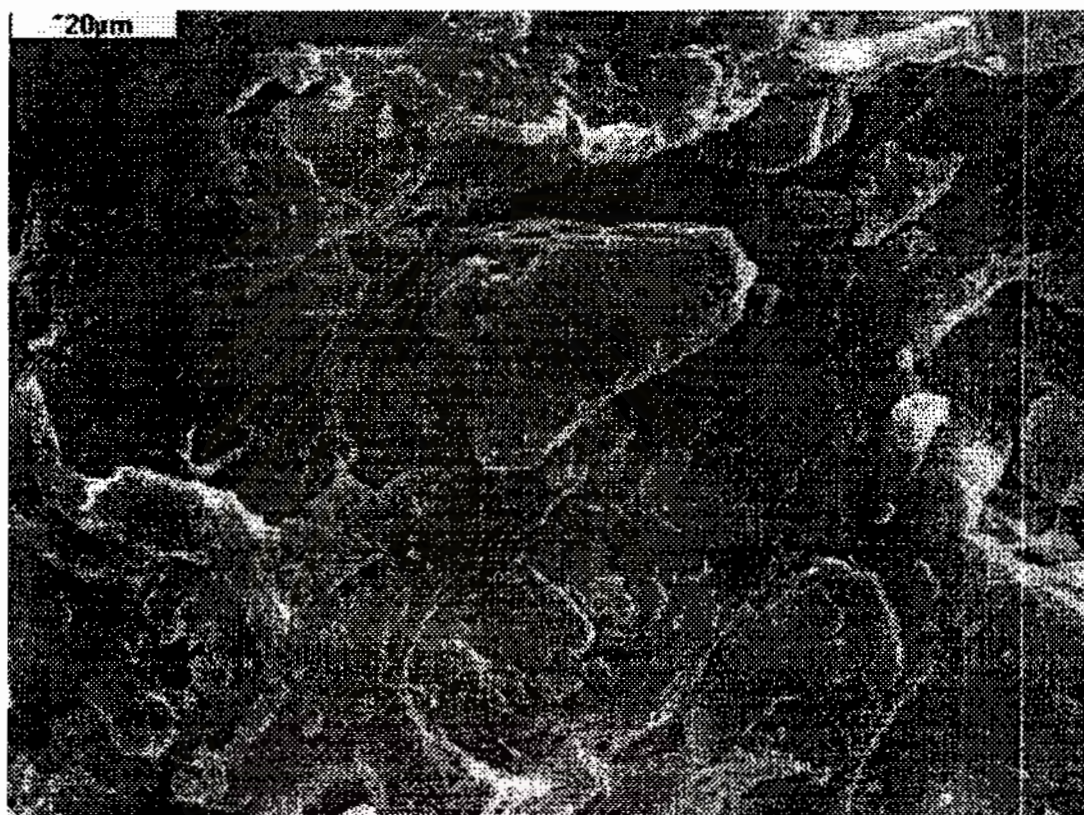


Figure 1 SEM micrograph of fracture surface of commercial resin

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Figure 2 SEM micrograph of fracture surface of N8038/JJ4324/CaCO₃:

30/5/65

จุฬาลงกรณ์มหาวิทยาลัย



Figure 3 SEM micrograph of fracture surface of N8038/JJ4324/Kaolin:

30/5/65

จุฬาลงกรณ์มหาวิทยาลัย



Figure 4 SEM micrograph of fracture surface of N8038/JJ4324/Talc: 30/5/65

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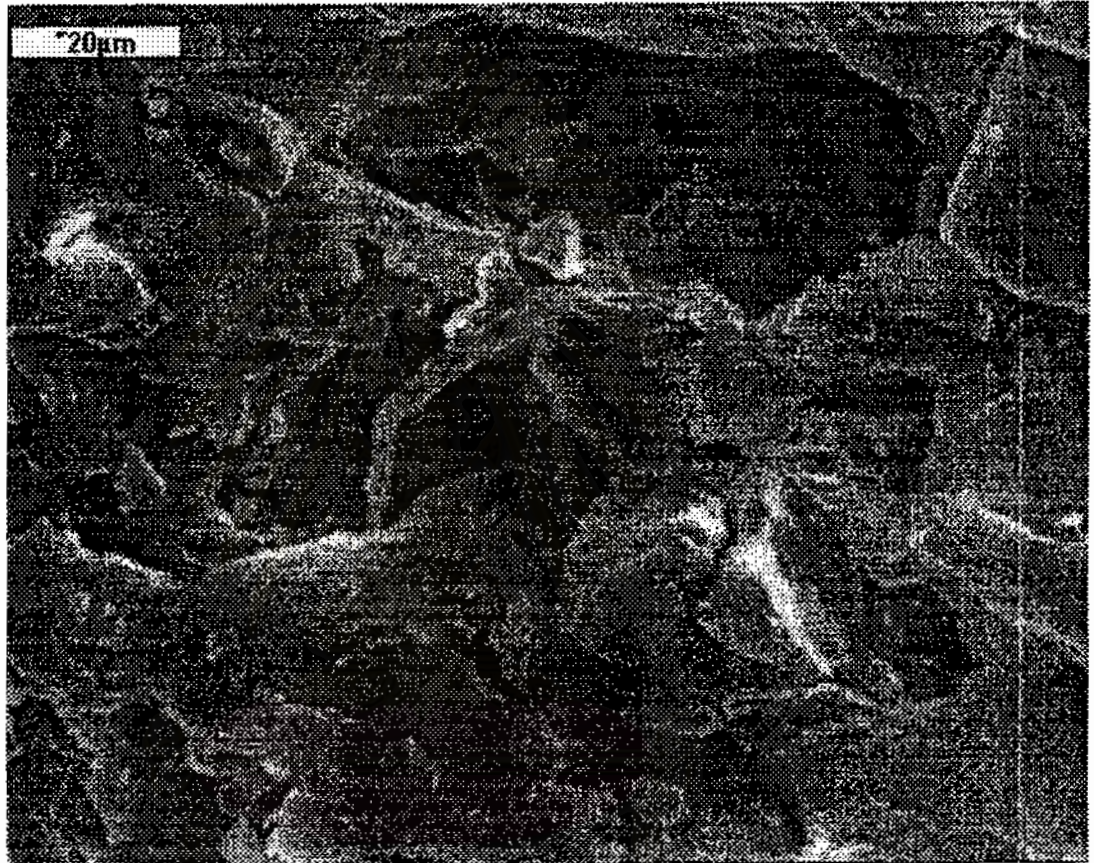


Figure 5 SEM micrograph of fracture surface of N8038/JJ4324/Talc: 25/5/70

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Figure 6 SEM micrograph of fracture surface of N8038/JJ4324/Talc: 20/5/75

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Figure 7 SEM micrograph of fracture surface of N8038/JJ4324/BaSO₄:

30/5/65

จุฬาลงกรณ์มหาวิทยาลัย



Figure 8 SEM micrograph of fracture surface of N8038/JJ4324/CaCO₃:

30/5/65

จุฬาลงกรณ์มหาวิทยาลัย

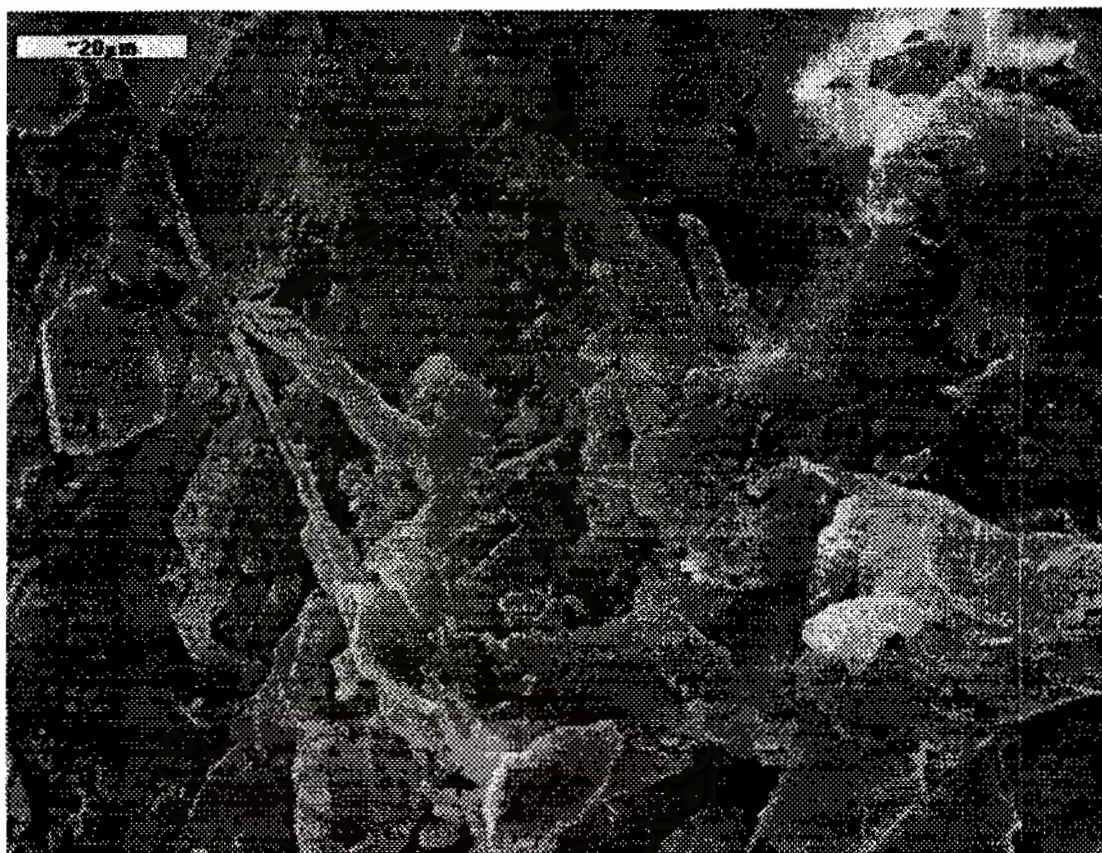


Figure 9 SEM micrograph of fracture surface of N8045/JJ4324/Kaolin:

30/5/65

จุฬาลงกรณ์มหาวิทยาลัย

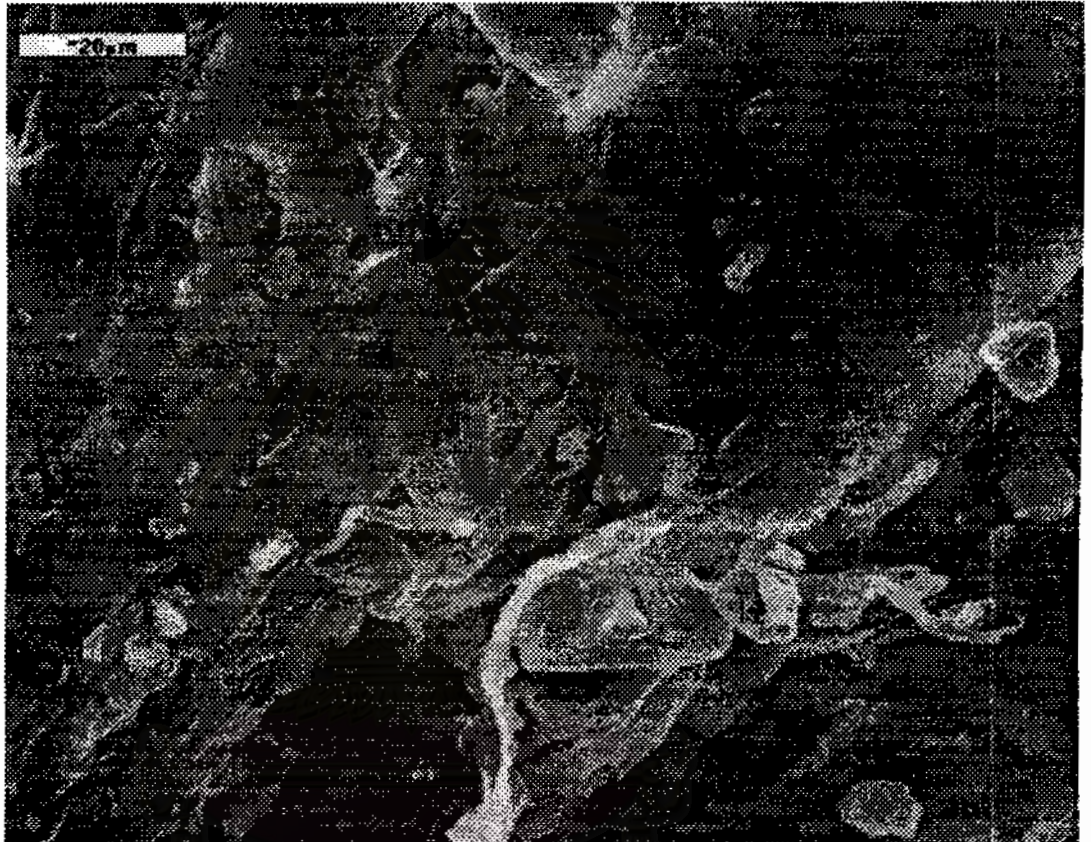


Figure 10 SEM micrograph of fracture surface of N8045/JJ4324/Talc: 30/5/65

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Figure 11 SEM micrograph of fracture surface of N8045/JJ4324/BaSO₄:

30/5/65

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