

CONTINUOUS VULCANIZATION OF EXTRUDED PROFILE BY MICROWAVE PROCESS

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ABSTRACT

Continuous vulcanization is being increasingly used today in the manufacture of extrusion profiles. This is particularly so with the microwave/hot air continuous vulcanization process. Although this process is now quite widely used in Europe and to a lesser extent in USA, it is still not used in Malaysia. To improve the technological capability of the rubber-based industry in extrusion product, the RRIM has acquired a microwave/hot air tunnel continuous vulcanization equipment to enable development work in this area to be carried out with the aim of upgrading the rubber industry towards this more automated manufacturing process. This is particularly pertinent in view of the anticipated labour shortage, and, increasing labour and energy cost.

This paper outlines the basic principles of operation of the microwave/hot air tunnel continuous vulcanization process and describes some aspects of compounding involving natural and synthetic rubbers for use in the process. As temperature increase is one of the major factors influencing the vulcanization of profile in this process, study was therefore concentrated on the heat generation aspect in the microwave tunnel.

ABSTRAK

Pervulkanan selangar sedang pesat digunakan pada masakini bagi membuat barangan daripada profil perlelehan. Ini ternyata terutamanya melalui proses gelombang mikro/pervulkanan udara paras. Walaupun pada masakini proses cara ini digunakan dengan meluas di Eropah berbanding dengan USA tetapi di Malaysia masih belum digunakan lagi. Untuk memperbaiki kebolehan teknologi bagi industri yang menggunakan getah dalam prosesan barangan perleleran, RRIM telah memperolehi satu alat gelombang mikro/pervulkanan udara panas untuk penyelidikan dengan tujuan meningkatkan lagi prestasi industri getah dalam menggunakan proses pengeluaran automatik. Ini lebih diperlukan memandangkan kemungkinan kekurangan pekerja dan kenaikan gaji pekerja dan kemahalan harga pengeluaran.

Kertas kerja ini menyalurkan cara asas penggunaan proses gelombang mikro/pervulkanan udara panas dan membentangkan aspek-aspek penyebatian menggunakan getah asli dan getah tiruan untuk digunakan dalam proses tersebut. Peningkatan suhu adalah salah satu faktor yang membawa kepada profil pervulkanan dalam proses ini, penyelidikan telah ditumpukan kepada penjaanahan haba didalam saluran gelombang mikro.

INTRODUCTION

The conventional batch process of producing rubber profiles involves firstly the extrusion of extrudates followed by vulcanization in an autoclave. This process therefore involves process handling which could cause distortion to extrudates. In addition, the batch process needs more labour input and has higher energy loss during in process transfer. In the continuous vulcanization process, shaping, vulcanization and finishing are all done in one single, continuous operation; therefore, there is little scrap generated, negligible energy loss between production steps and comparatively less manpower is needed (Capelle, 1988; Lue, 1980). Presently, there are several continuous vulcanization systems being used by this industry viz. vulcanization in hot air tunnel, liquid curing media or salt bath (Metherell), fluidised bed, and microwave or ultra high frequency vulcanization. There are many advantages of using the microwave process. Some of these are:

- o fast and clean operating procedure
- o production flexibility
- o rapid heating
- o energy saving
- o constant heating
- o relatively smaller floor space

Because of these, the microwave continuous vulcanization system is preferred over the other continuous vulcanization systems mentioned earlier.

Although continuous vulcanization systems are widely used in Europe, USA and Japan, it is new in this country particularly the microwave continuous vulcanization system. In view of labour shortage, and increasing labour and energy cost, the microwave process is particularly suitable for manufacturing of profile locally. To improve the technological capability of the rubber-based industry in extrusion product, the RRIM has recently acquired a microwave/hot air continuous vulcanization equipment with the IRPA fund to enable development work in this area to be carried out so as to assist the local industry in upgrading to this automated process. This paper describes some of the main features of the microwave continuous vulcanization system and some compounding aspects pertaining to this system.

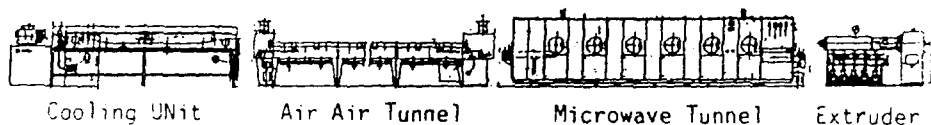
Equipment

A schematic diagram of the microwave continuous vulcanization process is shown in Figure 1. It consists of a vacuum extruder, microwave tunnel, hot air tunnel, cooling trough and take up conveyor and cutting equipment. The extruder used in the studies was a 90 mm vacuum extruder. It has a length to diameter ratio of 16:1. The vacuum system together with the usage of desiccant will ensure removal of volatile matters from the rubber compound. The volatile matters if not removed will cause porosities in rubber profile during vulcanization under atmospheric pressure. The extruder is equipped with heating/cooling system with automatic temperature control for screw, barrel and die head. This will ensure good control of compound temperature during extrusion. The microwave used in the studies has an available power of 10 kW. The microwave energy is generated by 4 magnetrons with a nominal rating of 3 kW each, but which are used at a

maximum of 2.5 kW. The power can be infinitely adjusted up to 10 kW. The tunnel has a total of 6 cavities with the first and last cavities being empty or not fitted with magnetron. Possible stray energy for adjacent cavities is absorbed into the profile. At the entry and exit of tunnel adjustable metal gates are fitted as wave traps for blocking any possible remnants of radiation. The cross section of the tunnel, 300 x 160 mm, allows most of the commonly used profile to be made. The tunnel which is 8.5 m in length is also conditioned with hot air at a temperature which is adjustable up to 200°C in order to ensure against possible radiation of heat generated with profile to the surrounding atmosphere. The conveyor belt is made of Teflon reinforced with glass fibre.

The hot air tunnel is 12 m in length. Heating of air is by electric heating elements and the hot air is distributed by ventilators. The belt used is the same as that in the microwave tunnel.

FIG.1 MICROWAVE/HOT AIR CONTINUOUS VULCANIZATION LINE



RESULTS

Comparison of microwave and hot air vulcanization

To assess the effect of using microwave energy in vulcanization, a comparison was made by extruding an NR compound through the microwave/hot air tunnel with and without applying microwave energy. The microwave energy of each magnetron was varied from 0.25 to 1 kW which means that the total energy for 4 magnetrons in the tunnel was varied from 1 to 4 kW. For the condition where no microwave energy was applied, the profile of cross section area of 4 x 50 mm was allowed to vulcanize in the hot air tunnel and samples were taken at various interval for determination of properties. The extrusion speed used was 3 m/min. Taking the total of the microwave/hot air tunnel as 20 metres, it means that the compound which has been subjected to microwave energy or preheating and vulcanization in the tunnel was 6.3 minutes. Figure 2 shows the variation in tensile strength and modulus of the NR compound with varying microwave energy input and Figure 3 shows the variation in tensile strength with vulcanization time in hot air tunnel without any microwave energy input.

FIG.2 TENSILE PROPERTIES VS MICROWAVE ENERGY

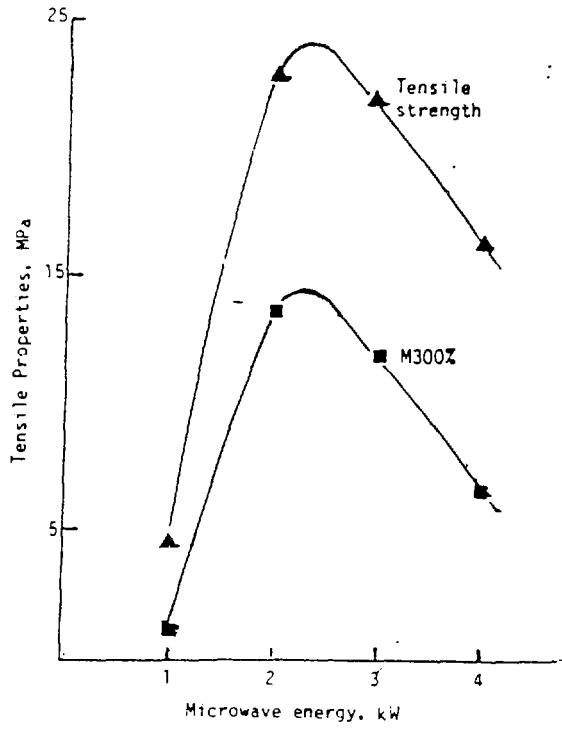
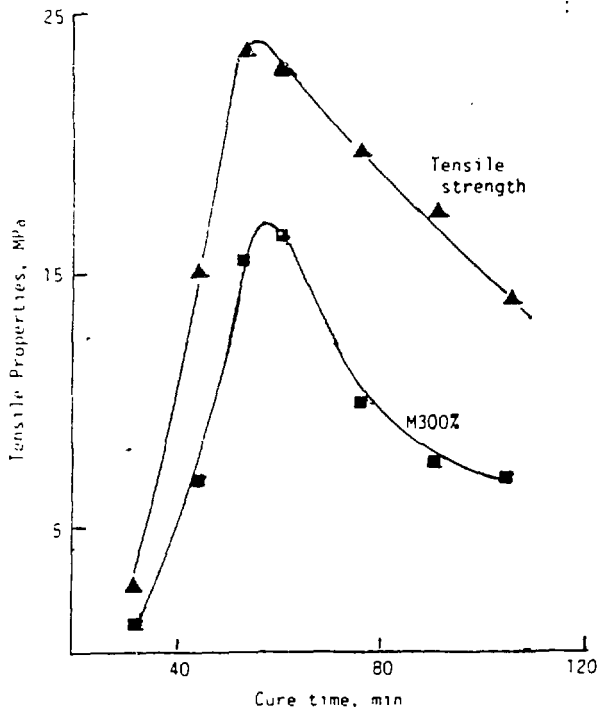


FIG.3 TENSILE PROPERTIES VS CURE TIME IN HOT AIR TUNNEL



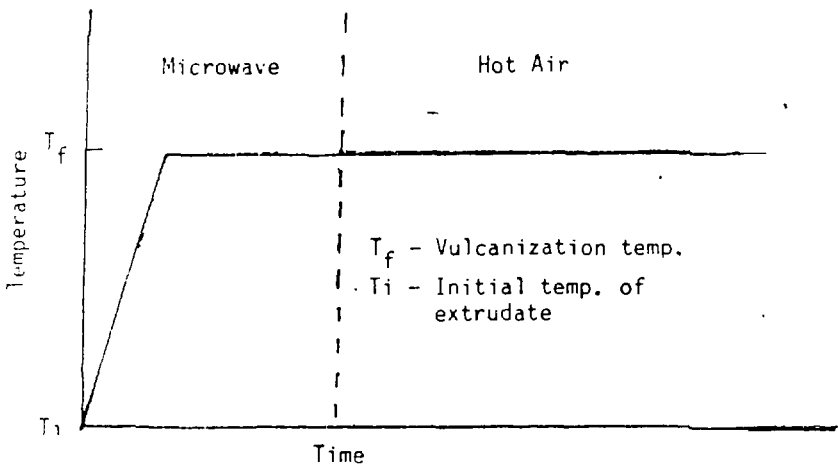
For the NR compound studied, the optimum level of microwave energy to achieve optimum tensile properties at 3 m/min extrusion rate was between 2 to 2.5 kW with the hot air temperature of 180°C. Below this optimum level of microwave energy, there is insufficient heat generation with the extrusion profile to effect vulcanization at 3 m/min while above this optimum level, reversion, as indicated by deterioration in properties, occurs. When no microwave energy was applied, the optimum tensile properties was achieved at a cure time of about 70 minutes which showed the inefficiency of hot air vulcanization. To improve the efficiency of this continuous vulcanization system, it is therefore important to raise the temperature of the profile to almost the vulcanization temperature in the microwave tunnel and then the profile is maintained at this temperature in the hot air tunnel until vulcanization is completed. In view of this, studies on the effects of compounding ingredients which affect the heat generation of compound in the microwave tunnel were carried out.

Effect of compounding ingredients on heat generation

(i) Rubber type

Figure 4 shows an ideal temperature profile of microwave/hot air continuous vulcanization system. The temperature of extrusion profile should be increased to the vulcanization temperature as quickly as possible before entering the hot air tunnel. Depending on the choice of compounding ingredients, different compounds affect the temperature increase differently.

FIG.4 IDEAL TEMPERATURE PROFILE



The choice of rubber type used in the rubber compound has significant influence of the heat generation. Table 1 shows the influence of rubber on temperature increase in microwave tunnel. Five gum compounds based on natural rubber (NR), polychloroprene (CR), acrylonitrile butadiene rubber (NBR), 34% ACN content, ethylene propylene diene monomer (EPDM) and epoxidised natural rubber (ENR 50) were extruded at 1.4 m/min through the microwave tunnel. The results show that the temperature increase follows the following order:

CR > NBR > ENR 50 > NR > EPDM.

TABLE 1 EFFECT OF RUBBER ON HEAT GENERATION IN MICROWAVE TUNNEL

	NR	CR	NBR	EPDM	ENR 50
Temp. at die, °C	122	122	127	128	121
Temp. at exit of microwave tunnel, °C	148	200	197	140	180
Temp. rise, °C	26	78	70	12	59

Microwave energy: 4 magnetrons, 0.5 kW each i.e. total energy input 2 kW
 Extrusion speed: 1 m/min
 Formulation: Rubber 100, ZnO 5, St. acid 1, Aromatic oil 5, CaO 5
 Die used: 18.7 mm diameter circular die

The increase in temperature is associated with the increase in polarity of the rubber. The effect of blending polychloroprene with natural rubber in heat generation in microwave tunnel was also investigated. Table 2 shows that increasing the proportion of polychloroprene rubber increases the heat generation of the gum compound.

TABLE 2 BLEND OF NATURAL RUBBER WITH POLYCHLOROPRENE

	NR	NR/CR 90/10	NR/CR 75/25	NR/CR 50/50	CR
Temp. at die, °C	122	122	125	125	125
Temp. at exit of microwave tunnel, °C	148	156	171	193	201
Temp. rise, °C	26	34	46	68	76

Microwave energy: 4 magnetrons, 0.5 kW each i.e. total energy input 2 kW
 Extrusion speed: 1 m/min
 Formulation: Rubber 100, ZnO 5, St. acid 1, Aromatic oil 5, CaO 5
 Die used: 18.7 mm diameter circular die

(ii) Fillers

The effect of four different types of carbon black of different particle sizes, viz. MT, FEF, HAF and ISAF blacks, in NR compounds was investigated. Figure 5 shows the relationship of temperature rise in an NR compound containing 50 pphr carbon black with the microwave energy. As the microwave energy increases, the heat generation in the extrudate increases. The larger particle MT black which has an average particle size of 270 nm gives lower heat generation when compared to the finer particle size black. However, as the differences in the particle size between FEF, HAF and ISAF (43, 28, 22 nm) are relatively small, the temperature rise between these black are relatively less. Figure 6 shows the effect of dosage of FEF in NR compound on heat generation in microwave tunnel. Increasing the dosage of black generally increases the heat generation.

FIG.5 EFFECT OF CARBON BLACK ON HEAT GENERATION

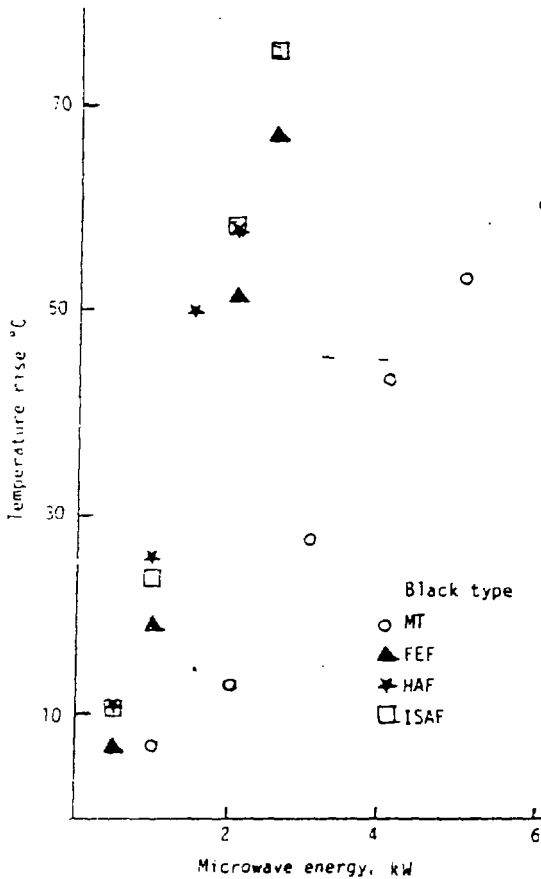
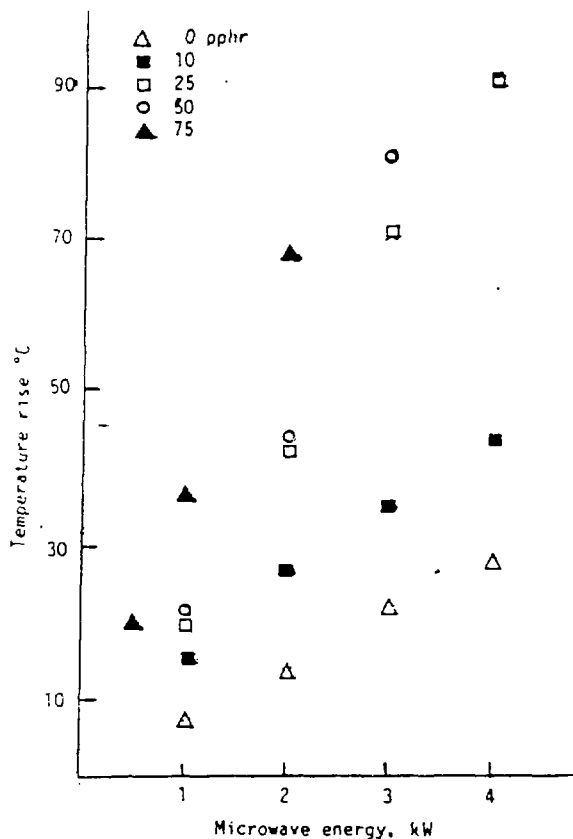


FIG.6 EFFECT OF BLACK LEVEL (FEF) ON HEAT GENERATION



The effect of non-black filler was also investigated. Table 3 shows the effect of four non-black fillers viz. ZnO, clay, whiting and silica at 50 pphr on heat generation in NR compound. Two levels of microwave energy at 2 and 4 kW were examined. As compared to black filler, the influence of non-black filler on temperature increase in the compound was significantly lower. Among the non-black filler investigated, ZnO gave the largest increase in temperature particularly at higher microwave energy level of 4 kW.

**TABLE 3 EFFECT OF NON BLACK FILLER ON HEAT GENERATION
IN MICROWAVE TUNNEL**

Filler	ZnO		clay		whiting		silica	
	2	4	2	4	2	4	2	4
Microwave energy, kW								
Temp. at die, °C	120	120	119	120	128	128	150	150
Temp. at exit of microwave tunnel, °C	148	172	144	158	136	148	163	188
ΔT, °C	28	62	25	38	8	20	13	38

Formulation: NR 100, ZnO 5, St. acid 1, Aromatic oil 5, CaO 5, Filler 50
Die used: 18.7 mm diameter circular die

Effect of extrusion rate on heat generation

Table 4 gives the temperature rise of extrusion profile at 3 different extrusion rates. The temperature rise in a black loaded EPDM compound was measured using a container seal die. As expected the faster the extrusion rate the lower the temperature increase in the profile. The results also show that for the EPDM compound requires double the microwave energy to compensate for the 3 times increase in extrusion rate.

TABLE 4 EFFECT OF EXTRUSION RATE ON HEAT GENERATION

	Extrusion rate m/min			
	1.3	2.6	3.9	3.9
Microwave energy, kW	2	2	2	4
Screw speed, rpm	13	26	40	40
Temp. at die, °C	100	106	112	121
Temp. at exit of microwave tunnel, °C	156	145	140	176
Temp. rise, °C	56	39	28	55

Formulation: EPDM 100, ZnO 5, St. acid 1, N550 120, Aromatic oil 80,
whiting 30, CaO 5
Die used: C die

Comparison of microwave/hot air vulcanization and compression moulding

Comparison between microwave/hot air vulcanization and compression moulding were carried on four compounds based on NR, NR/CR blend, ENR 50 and NBR (27% ACN content). The properties obtained by the microwave/hot air vulcanization were obtained at optimum level of microwave energy. Tables 5-8 show the tensile and hardness properties obtained by the microwave/hot air process at optimum dosage of microwave energy and the compression moulding at optimum cure time, as assessed by a Monsanto Rheometer at 180°C. In all the four compounds examined, compression moulded samples show slight differences in vulcanizate properties as compared to the microwave/hot air samples. The compression moulded samples tend to give slightly higher tensile strength and hardness.

TABLE 5 MICROWAVE/HOT AIR VULCANIZATION VS COMPRESSION MOULDING

SMR CV	100	
ZnO	5	
St. acid	2	
N550	50	
Aromatic oil	5	
Microcrystalline wax	1	
6PPD	2	
CBS	2	
TMTD	0.2	
S	1	
PVI	0.1	
CaO	5	
Microwave/hot air cure		Press cure
0.5 kW x 4/180°C		2.5'/180°C
Line speed 3m/min		
Tensile strength, MPa	22.8	23.9
Elongation at break, %	467	443
M300%, MPa	13.54	14.99
Hardness, IRHD	57.0	59.5

TABLE 6 MICROWAVE/HOT AIR VULCANIZATION VS COMPRESSION MOULDING

Neoprene W	40	
N774	5	
Whiting	80	
Aromatic oil	10	
Microcrystalline wax	1	
6PDD	0.5	
MgO	4	
ZnO	5	
S	0.5	
Ethylene thiourea	2	
CaO	5	
Microwave/hot air cure		Press cure
0.75 kW x 4/180°C		2.5'/180°C
Line speed 3 m/min		
Tensile strength, MPa	5.9	6.3
Elongation at break, %	547	443
M300%, MPa	2.02	3.02
Hardness, IRHD	42.0	49.5

TABLE 7 MICROWAVE/HOT AIR VULCANIZATION VS COMPRESSION MOULDING

ENR 50	100	
ZnO	5	
St. acid	2	
N550	50	
Aromatic oil	5	
6PDD	2	
TMTD	0.2	
CBS	2	
S	1	
PVI	0.5	
CaO	5	
Microwave/hot air cure		Press cure
0.25 kW x 4/180°C		2.5'/180°C
Line speed 3 m/min		
Tensile strength, MPa	18.6	21
Elongation at break, %	313	403
M300%, MPa	17.6	15.87
Hardness, IRHD	65.5	67
Compression set	12.0	9.5
72 hr/23°C, %		

TABLE 8 MICROWAVE/HOT AIR VULCANIZATION VS COMPRESSION MouldING

NBR (27% ACN content)	100	
ZnO	5	
St. acid	1	
N774	70	
Aromatic oil	15	
6MLD	2	
CBC	1	
TMTD	0.4	
S	2	
CaO	5	
Microwave/hot air cure		Press cure
0.5 kW x 4/180°C		2.0'/180°C
Line speed 3 m/min		
Tensile strength, MPa	16.0	18.2
Elongation at break, %	331	449
M300%, MPa	13.2	12.75
Hardness, IRHD	64.5	64.5

CONCLUSION

Application of microwave energy has been shown to accelerate the vulcanization process as compared to hot air vulcanization. In the microwave/hot air continuous vulcanization process, it is important to raise the temperature of a profile to almost the vulcanization temperature in the microwave tunnel so as to increase the efficiency of the process.

The increase in the temperature of the profile has shown to be dependent on the compounding ingredients and extrusion speed. Polar rubbers generate more heat in the profile as compared to non-polar rubbers. Finer particle size carbon black gives higher heat generation as compared to the larger particle size black. Non-black fillers are less effective in this respect as compared to carbon black.

Some of the basic vulcanizate properties of profile obtained by the microwave/hot air vulcanization and compression moulding processes were compared. There were slight differences in the properties obtained at optimum cure conditions between the two processes.

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