

SELECTIVE BLEND OF EPDM-VMQ RUBBER FOR HIGH HEAT RESISTANT APPLICATION⁺

خلطة مطاطية مختارة من مركب (EPDM-VMQ) للاستخدام كمادة ذات مقاومة حرارية عالية

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Abstract:

For some applications it is usually recommended to have high heat resistant elastomer materials. Compounds with blend of EPDM (Ethylene-Propylene Rubber) and VMQ (Dimethyl polysiloxane Rubber) or silicone rubber of medium and high VA (Vinyl Acetate is 45% and 70% in VMQ) along with ingredients are used in this paper to fulfill high heat resistant compound. Synthetic fabric was used as reinforcing agent with the rubber compounds. The physico-mechanical properties, rheometric characteristics, abrasion resistance and adhesion property with different textile members were studied for this selective blend. Ageing study was carried out on a vulcanized samples. Overall properties of EPDM/VMQ blends based on VMQ with high AV content are found to be better compared to blends based on VMQ with medium VA content.

KEY WORDS: VMQ of VA (Vinyl Acetate), Physico-mechanical properties, Rheometric Characteristics, and Ageing), RFL(Rubberized Fabric Layers).

المستخلص :

توصف المواد المطاطية ذات المقاومة الحرارية العالية عادة، عندما تكون هناك حاجة لبعض الاستخدامات في ظروف تكون عندها الدرجات الحرارية مرتفعة، وقد تم في هذا البحث استخدام مركب مطاطي مؤلف من مادتين هما مطاط الاثيلين بروبيلين EPDM ومطاط السليكون VMQ المتوسط التركيز والعالي التركيز لمادة خلات الفايثيل (VA (Vinyl Acetate are either 45% or 70% in VMQ) ، إضافة إلى بقية المواد الأخرى المستخدمة في البحث والتي تؤمن الحصول على خلطة مطاطية مقاومة للحرارة. استخدم أيضا عامل تقوية مؤلف من ألياف نسيجية صناعية لتقوية مركب المطاط. تم في البحث اختيار خلطة مطاطية ودراسة الخصائص العامة لها بعد الفلكنة، مثل الخصائص الريومترية Rheometric Characteristics ، والخواص الفيزيوميكانيكية physico-mechanical ، وخاصة مقاومة البري أو الحك Abrasion Resistance ، وخاصة الالتصاق adhesion للشبكات النسيجية المختلفة والمستخدم لإغراض التقوية مع عجينة المطاط. كما تمت دراسة عملية الازمان أو التقادم Ageing study لعينات الخلطة نفسها. إجمالي الخواص لمركب مطاط الـ EPDM/VMQ اخذت على أساس استخدام مطاط السليكون VMQ ذو الفايثيل اسيتيت (خلات الفايثيل) بتركيز 70% والذي اظهر تحسنا بالخواص نسبة إلى مطاط السليكون الذي يحوي خلات الفايثيل بتركيز 45%.

INTRODUCTION:

There are different types of elastomer available for usage in high heat resistant compound. These types of elastomer are used alone or after blending with two or more of such elastomer

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for high heat resistant applications. These types of elastomer are generally known as thermo-stable elastomer. These are viz., VMQ, ACM(Polyacrylate Rubber), CSM(Chloro-sulphonyl polyethylene rubber), HNBR(Nitrile Rubber), FBM(Viton Fluorocarbon), CIIR(Chlorinated Butyl Rubber), BIIR(Brominated Butyl Rubber), etc. But such type of elastomer has certain limitation from processing problem. Desired modification in the compound design along with other input ingredients are utilized to make it suitable for high heat resistant applications[1].

Presently to withstand a temperature for 150°C, heat resistant compound having blend of EPDM/BIIR or EPDM/CIIR are used but these blended compounds have some inherent processing problem. Compounds with halogenated butyl rubber having high air impermeability generally causes processing problem due to formation of blisters during vulcanization of the finished product. Moreover, it also gives rise to adhesion problem with synthetic textile reinforcing member in a rubber composite due to its non-polar characteristics[2].

In view of this, it is necessary to develop a suitable formulation either with single or blend of two or more elastomer for designing of suitable heat resistant compound which may withstand a fluctuated temperature of around 200°C at the application point. For high heat resistant application, compound may be used with the blend of EPDM and VMQ along with other ingredients[3]. As it is known for the chemists silicon rubber VMQ consists of long chains of alternating silicon-oxygen atoms encased by organic groups. High temperature resistance for silicon rubber is mainly related to covalent bonds. These chemical bonds are found in other high temperature materials, such as quartz, glass, and sand. The addition of Vinyl side-chains $[-CH=CH_2]$ improves vulcanization characteristics and heat ageing[4]. Without addition of Vinyl acetate the physical and mechanical properties of the blend of EPDM/VMQ had already been studied by other researchers [5]. In this paper, although the same blend of EPDM/VMQ has been selected but compound optimization has been done by changing various compound ingredients so that it becomes compatible with dipped RFL(Rubberized Fabric Layers) such as Vinyl Acetate content for high heat resistant application i.e., at around 200°C.

Moreover for doing this study, the effect of Vinyl Acetate content (either 45% or 70% by weight) in VMQ rubber composition with EPDM rubber has also been measured and specified to ascertain the heat resistant characteristics.

EXPERIMENTAL:

Compound formulations both for cover and skim coat were made based on blends containing EPDM and VMQ rubber with medium (45%) and high (70%) Vinyl Acetate content.

All compounds were prepared on a two roll mixing mill (33x15 cm) at a friction ratio of 1:1.25 maintained at 70±5°C temperature. Curing characteristics at 170°C were measured on Monsanto Rheometer R-2000 with accordance to ASTM D2084-89[6].

Physico-mechanical properties were measured on the cured test specimens as per ASTM standard methods. These are viz., Tensile Strength, Modulus and Elongation were tested with accordance to ASTM D412-88, Shore Hardness was tested with accordance to ASTM D1415-89, Abrasion loss with accordance to ASTM D2084-89, Adhesion test was carried out according to British Specification (B.S 903-PTA9) [7]. Ageing study was carried out on the vulcanized compound in a cell type oven at temperature of 170°C for 72 hrs.

The peel adhesion test of synthetic fabric with the rubber compounds was done according to the modified ASTM D2630-71 method. RFL(Rubberized Fabric Layers), treated synthetic fabric was rubberized both sides with skim rubber compound at a uniform thickness of 0.4mm in a calendar machine. The top and cover compounds were sheeted out of from two

roll mill with uniform thickness of 3mm. The composite was prepared by lying three rubberized synthetic fabrics one over the other. The top and bottom covers were then applied and the whole assembled composite was cured in a heated press at temperature of 170°C. The peel adhesion strength between rubber to fabric and also between fabric to fabric was determined by using a tensile testing machine. In this paper the need for using reinforcement rubber sheets of RFL was requested by the Iraqi Southern Cement Company for attempting manufacturing conveyer belt could be used at elevated temperature medium in Kufa cement plant.

In the present study two different experimental composite product samples were used, first one of type **A** associated with formulation of the **mix1** for the cover and skim of the peel and second one of type **B** associated with formulation of **mix2** also for the cover and skim of the peel. Those samples were prepared and peel adhesion strength both in the longitudinal and transverse directions were measured after cutting test samples. Heat aged peel strength was measured after ageing the pad samples at 170°C for 72hrs.

RESULTS:

Compound formulations for both cover and skim compound used in present study were illustrated in **table-1**.

The rheometric characteristics of the compounded rubber were shown in **table-2**.

The physico-mechanical properties including heat ageing tests at various temperatures were measured and the test results were given in **table-3**.

The peel adhesion strength of the compounded rubber with RFL dipped synthetic fabric both unaged and heat aged conditions at certain ageing temperature(170°C) were measured and test results were reported in **table-4**.

Table-1: Formulation of the Mixes

INGREDIENTS USED	MIX CODE			
	Mix No.1 A _{cov}	Mix No.2 A _{skim}	Mix No.1 B _{cov}	Mix No.2 B _{skim}
EPDM(elastomer)	60	40	60	40
VMQ(45%VA) (elastomer)	40	60	----	----
VMQ (70%VA) (elastomer)	----	----	40	60
Stearic acid(accelerator activator)	0.5	0.5	0.5	0.5
Zn-streate(metal accelerator activator)	----	0.5	----	0.5
HAF (high abrasion furnace)carbon black	50	30	50	30
Vulkanox FH(miscellaneous chemicals)	5	5	5	5
Spindle oil (processing aids)	----	2	----	2
SP-80(anti-oxidant)	5	----	5	----
MC-Wax(protective wax)	3	----	3	----
Accinox BL(high temp.anti-ozonant)	2	----	2	----
Accinox TQ (high temperature anti-oxidant/anti-ozonant)	1	1	1	1
DCP(vulcanizing agent)	6	4	6	4
TAC(vulcanizing co- agent)	2.5	3	2.5	3

Table-2: Cure Characteristics of the Mixes.

Cure Properties	MIX CODE			
	Mix No.1 A_{cov}	Mix No.2 A_{skim}	Mix No.1 B_{cov}	Mix No.2 B_{skim}
Min. torque(M_I),dN.m	16	8	18	9
Max. torque(M_{max}),dN.m	56	40	47	38
Induction time (t_{S1}), min.	1.96	2.25	2.27	2.20
Scorch time (t_{S2}), min.	2.02	2.40	2.38	2.66
Optimum cure time (t_{90}), min.	18.20	15.50	12.00	12.00
Cure rate index (min^{-1})	6.18	7.63	10.40	10.71

Table-3: Physico-mechanical Properties of the Mixes

Property tested	MIX CODE				
		Mix No.1 A_{cov}	Mix No.2 A_{skim}	Mix No.1 B_{cov}	Mix No.2 B_{skim}
Surface Tack, gm		Moderate	Good	Fair	Good
Abrasion Index		112	---	110	---
Modulus at 300% elongation, Kg/cm ²	Unaged	84.36	28.56	76.14	39.87
	Aged ¹	64.45	25.00	63.27	42.58
Tensile Strength, Kg/cm ²	Unaged	115.24	77.63	123.58	88.78
	Aged	74.33	64.28	85.59	84.87
Elongation at break, %	Unaged	480	750	550	608
	Aged	358	600	496	585
Shore Hardness	Unaged	63.24	52.53	62.63	55.56
	Aged	78	61	76	64

Aged¹= Heat Aged At 170°C for 72 hrs.

Table-4: peel Adhesion (Kg/25mm)

Sample	Direction of Peeling	Cover to Ply		Ply to Ply	
		Unaged	Heat Aged ¹	Unaged	Heat Aged ¹
A	Longitudinal	11.0	13.0	13.5	14.5
	Transverse	10.5	14.0	11.2	13.6
B	Longitudinal	12.5	14.7	15.5	18.0
	Transverse	10.8	14.0	13.1	16.5

Aged¹= Heat Aged At 170°C for 72 hrs.

All data of percent change of physical properties and peel adhesion strength on heat ageing tests were also presented in table-3 and table-4.

DISCUSSION:

Based on previous studies on different blends compositions of EPDM and EPDM/VMQ, [1, 8,9,10]. 60/40 blend ratio for cover rubber compound and 40/60 blend ratio for skim rubber compound were selected. These blend compositions for both cover rubber and skim rubber compound exhibited balanced physico-mechanical properties for making super heat resistant composites.

In order to find the effect of VA content in VMQ rubber on the performance in the blended rubber compound with EPDM, two different grade of VMQ, one (45%VA content) and another (70%VA content) were selected for making both cover and skim rubber compounds.

For saturated polymers as in this blend of EPDM/VMQ, peroxide curing system was utilized together with a co-agent as per normal practice. In our present study, 4 to 6 pphr of dicumyl peroxide (DCP) and 2 to 3 pphr of triary cyanurate (TAC) were used for making both the compounds.

A- CHANGE IN CURE CHARACTERISTICS

The rheometric test results in table-2 reveal that the cure characteristics of compounds change quite appreciably with the change in grade of VMQ rubber in the blend with EPDM rubber for both covers (A_{cov} & B_{cov}) and skim (A_{skim} & B_{skim}) compounds. Cover (B_{cov}) and skim (B_{skim}) compounds made with VMQ rubber having high VA content (70%) shows slightly delayed induction and scorch time and quick cure time compared to compounds made with VMQ of moderate (45%) VA content, this may referred to increasing of VA content working as a retarding agent for accelerators and accelerator activators in the beginning of reaction at normal temperature and be a catalyst with increasing of reaction temperature, moreover curing with peroxide system i.e "Vinyl Specific" peroxides resulting in a such optimization of rheometric properties[11].

B- COMPARISON OF PHYSICO-MECHANICAL PROPERTIES

It can be seen from the physico-mechanical properties of the compounded rubber that the unaged properties of blends for both cover and skim compounds generally meet all the requisite criteria including sufficient surface tack for making laboratory scale rubber composite sample which could be used for high heat resistant application, and the same observations are noticed in case of blended compounds made with both grades of VMQ rubber.

The analysis of the heat ageing tests at certain temperature shows table-3 a significant improvement in retained physical properties for both cover as well as skim compounds when blended rubber was made with VMQ of high VA content compared to rubber blended with VMQ of medium VA content. This may be referred to the replacement of the methyl groups by vinyl acetate groups in VMQ which may give the most probable molecular weight distribution which is responsible for that improvement[4].

C- COMPARISON OF PEEL ADSHESION PROPERTIES

If the peel adhesion strength values are considered between rubber composite of type A (made with VMQ compound with medium VA content) and type B (made with VMQ compound with high VA content), it can be seen that there is an appreciable improvement in adhesion strength for both between cover rubber to ply and between ply to ply for compounds containing VMQ with high VA content compared to compounds with VMQ of medium VA content.

However after heat ageing test, a moderate to high increase in peel adhesion strength is noticed at the ageing temperature and also in case of both grades of VMQ rubber. This clearly indicates that post curing of the compounds has increased the values of all the properties, because as the vinyl acetate content of the vulcanizates increases heat aging increases due mainly to random structure[12].

As mentioned above most of the improvements in properties are taking part mainly due to Vinyl-containing silicones with vulcanizing system of peroxide, because it appears that the radicals derived from peroxide are able to add to vinyl groups and so give radicals which then form crosslinks by addition to second radical site, or by interaction with methyl side chain of a second rubber molecule with the formation of a second radical which can then promote further crosslinking[13].

CONCLUSION:

The retained physico-mechanical properties and also adhesion strength of the rubber compounds after high temperature ageing tests are greatly influenced with the introduction of VMQ rubber in blends with EPDM rubber. The other parameters viz., surface tack of the skim compound, process ability of the compound in the mill etc., also demonstrate the suitability of this blended rubber compounds for making a desired rubber composite. The above test results also confirm that all the properties of the blended compounds may be enhanced further with an increase in VA content in VMQ rubber.

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