

EVALUATION OF SOME INDIGENOUS MINERALS AS FILLERS FOR THE RUBBER INDUSTRY

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ABSTRACT

Fillers have played a major and a versatile role in the rubber industry with respect particularly to the reinforcement in vulcanized rubber products and cheapening of rubber compounds. In Sri Lanka we have numerous mineral resources which can replace some of those expensive imported fillers. The substitution of imported material to a greater extent would depend on the cost performance ratio of the local substitutes.

This paper deals with some work carried out using local white (non-black) mineral powders supplied by Messrs Lanka Wall Tiles Limited of Balangoda. The study of their performance in our trials compare well with conventional imported fillers and shows properties on par with the latter, particularly in the case of calcite and kaolinite. The test results of the physical properties of the vulcanized rubber compounds, prepared, using each of the above fillers, as well as their individual properties are discussed in this work.

INTRODUCTION

Six common mineral powders available locally at Lanka Wall Tiles Limited were selected for these trials. All of these minerals are found as ore deposits distributed in hilly areas along with laterite soil. They are namely quartz, silica sand, calcite, kaolinite, feldspar and ground tile (powdered defective tiles).

Of these, quartz and silica sand are of silica origin (SiO_2), calcite is a naturally occurring form of calcium carbonate (CaCO_3) and kaolinite is a hydrated form of aluminium silicate ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$). Tile powder is a mixture of aluminium silicates (silica common clay) and feldspar is a mixture of sodium, potassium, calcium and aluminium silicates.

In our trials, the indigenous silica fillers were compared with a precipitated reinforcing silica (Vulkasil S) which is a conventional silicate filler commonly used in the rubber industry. Calcite was compared with refined precipitated calcium carbonate (Winnofil S) and kaolinite feldspar and tile powder with another common conventional filler, kaolin.

The process of manufacture of these mineral powders consist of two stages. *i.e.*

Dry calcining stage of the ore followed by..

A grinding process in ball-mills and vibro-mills.

At the Balangoda Wall Tile Factory these minerals are produced to meet the demands of the local ceramics industry. Calcining and ball-milling treatments endow the resulting

powders with properties of low-moisture content and fine particle size both of which are important physical parameters sought after in rubber fillers.

EXPERIMENTAL

In the determination of moisture content a ten gram (10 g) sample of the filler was heated at 105°C in an oven and reweighed. The difference in weights was taken as a measure of moisture content.

Particle size was determined using a 160 mesh sieve (98 millimicrons) to sift the powders after drying them at 100°C. The results are given as a weight percentage of the particles that passed through the sieve without disturbing the agglomerates of powdered filler.

In compounding the following basic formulation was used to determine the amount of reinforcement imparted by the fillers.

Natural rubber	100 ms.
Stearic acid	1.5 g. or pphr.
Zinc oxide	5.0 " " "
CBS(accelerator)	1.2 " " "
IPPD(antidegradant)	1.0 " " "
Sulphur	2.5 " " "
Filler	30.0 " " "
Diethylene glycol	2.0 (only with silica fillers).

RESULTS AND OBSERVATIONS

Moisture content and the particle sizes of fillers under trial are compared with those of conventional ones in Tables 1 and 2 respectively.

Table 1. *Moisture content of fillers*

<i>Filler</i>	<i>Moisture %</i>
<i>Silica group</i>	
(1) Silica sand	0.04
(2) Quartz	0.05
(3) <i>Vulkasil S</i> (pptd reinforcing silica)	0.05
<i>whiting group</i>	
(1) Calcite	0.4
(2) <i>Winnofil S</i>	0.4
<i>Kaolin group</i>	
(1) Kaolinite	0.7
(2) Feldspar	0.1
(3) Ground tile	9.0
(4) <i>Kaolin</i>	0.5

From Table 1 it can be seen that the moisture content of indigenous fillers are on par with their counterparts except for tile powder.

Table 2. *Particle size*

<i>Filler</i>	<i>% Under 98 mm</i>
<i>Silica group</i>	
(1) Silica sand	88·0
(2) Quartz	70·0
(3) <i>Vulkasil S</i>	95·0
<i>Whiting group</i>	
(1) Calcite	77·0
(2) <i>Winnofil S</i>	85·0
<i>Kaolin group</i>	
(1) Kaolinite	59·0
(2) Feldspar	86·0
(3) Ground tile	77·0
(4) <i>Kaolin</i>	88·0

However it was noted that Kaolinite which gave 59% of particles under 98 microns in agglomerate form, increased the fine particle content to 85% when lightly rolled in a mortar with a pestle. Particle size determinations show that the local fillers were slightly coarser than the conventional fillers. In carrying out the above test great care was taken not to disturb the original agglomerate form.

The pH values of these fillers are given in the Table 3.

Table 3. *pH values of filler*

<i>Filler</i>	<i>pH</i>
<i>Silica group</i>	
(1) Silica sand	7·3
(2) Quartz	6·6
(3) <i>Vulkasil S</i>	6·4
<i>Whiting group</i>	
(1) Calcite	8·0
(2) <i>Winnofil S</i>	8·3
<i>Kaolin group</i>	
(1) Kaolinite	5·8
(2) Feldspar	7·2
(3) Ground tile	7·2
(4) <i>Kaolin</i>	5·8

A glance at the Table 3 shows that silica fillers are almost neutral, calcite and winnofil are basic, while kaolin and kaolinite are slightly acidic. Feldspar and tile powder also are almost neutral in reaction.

Compounding

During compounding (mixing) of the batches, according to the formulation given previously, following observations were made in connection with the case of incorporation of trial fillers.

Of the silica group Vulkasil S was the most difficult to be mixed while quartz and silica sand mixed in easily. Mixing Winnofil S was slightly difficult in comparison to calcite which was easily mixed. All others were incorporated quite easily.

In the case of silica fillers, diethylene glycol was used, at 2 pphr as an activator in later trials, while they were studied without activator in preliminary trials.

Table 4. *Cure characteristics*

<i>Fill</i>	<i>Cure time 100% cure mins</i>	<i>Scorch time mins</i>	<i>Induction period mins</i>
Quartz (unactivated)	22	4½	3½
Silica sand („)	18½	6¾	5½
Vulkasil S („)	65	14	10
Vulkasil S (activated)	22	4½	3½
Calcite	22	7½	4½
Winnofil S	20	6	4½
Feldspar	22	5	4
Kaolinite	19	4½	3½
Kaolin	22½	6	4½

VULCANIZATE PROPERTIES

The specific gravities were determined for the vulcanizates and are given along with the specific gravities of respective filler (Table 5).

Each compound was vulcanized for its respective cure time and the resulting vulcanizates were tested for physical properties, such as tensile strength, modulus, hardness, resilience, etc.

Table 5. *Specific gravity values*

<i>Filler</i>	<i>After compounding</i>	<i>Individual filler</i>
<i>Silica group</i>		
Quartz	1.12	2.4
Silica sand	1.11	2.3
Vulkasil S	1.08	2.0
<i>Whiting group</i>		
Calcite	1.1	2.4
Winnofil	1.11	2.5
<i>Kaolin group</i>		
Feldspar	1.1	2.6
Tile powder	1.1	2.3
Kaolinite	1.11	2.6
Kaolin	1.1	2.5

Physical properties under consideration are those that the filler would produce a significant effect at 30 pphr. Tensile strength was tested after maturing for 24 hours initially and followed up by testing the same vulcanized sheets after an year of shelf ageing. They were then aged for a further period of 48 hours at 70°C in an oven and tested.

Table 6. *Physical properties of vulcanizates each filler*

<i>Filler</i>	<i>Hardness IRHD degrees</i>	<i>Abrasion resistance volume loss cc/hr Dupont</i>	<i>De Mattic Flex Kc to Grade C</i>
Quartz	46	13.5	38.3
Silica sand	45	13.5	38.5
Vulkasil S	58	15.0	300.0
Calcite	45	14.4	198.0
Winnofil S	46	15.1	144.0
Feldspar	40	13.6	—
Tile powder	42	16.3	—
Kaolinite	47	15.8	138.0
Kaolin	46	13.7	150.2

Table 7. *Tensile strength of vulcanizates*

<i>Filler</i>	<i>T. S. unaged kg/cm²</i>	<i>T. S. after one year kg/cm²</i>	<i>T. S. after ageing for 48 hours kg/cm²</i>
	(EB)	(EB)	(EB)
Vulkasil S - activated	231. (at 700%)	302.(625)	227.(625)
Vulkasil S - normal	152. (at 650%)	243.(725)	259.(575)
Silica sand - activated	158. (at 550%)	201.(550)	149.(500)
Silica sand - normal	163. (at 575%)	203.(575)	170.(500)
Quartz powder	170. (at 600%)	152.(550)	135.(450)
Calcite	149. (at 600%)	187.(600)	161.(550)
Winnofil S	166. (at 600%)	174.(550)	135.(500)
Kaolinite	211. (at 600%)	262.(600)	201.(550)
Feldspar	53. (at 600%)	264.(600)	202.(550)
Tile powder	64.20(550)	—	—
Kaolin	220. (at 600%)	225.(575)	175.(500)

DISCUSSION

The comparatively high incidence of particles over 98 millimicrons in the case of kaolinite may not be the real value since it was in a state of agglomeration, since all the fillers were handled carefully so that original state was kept undisturbed. But in essence what takes place in the mixing of rubber and filler together in a 2-roll mill is a direct dispersion of these agglomerates due to frictional breakdown. Therefore the particle size should be still lower for all fillers under trial.

In the silica group all three fillers were activated with diethylene glycol along with 2% anhydrous calcium oxide to remove any trace of moisture that may be present in the activator. But this activation did not produce any significant effect on the physical properties of silica sand and quartz vulcanizates.

Tile powder and feldspar showed low hardness and tensile properties in the preliminary trials indicating poor reinforcement. Consequently they were screened out as failures from further trials.

The tensile properties clearly shows that there is an increase of tensile strength due to an optimisation of curing of the samples on maturation of the vulcanizates for one year which is also termed shelf-ageing. It is interesting to note the significant increase of tensile strength in the second column with the exception of quartz-based vulcanizates.

CONCLUSION

Of the six indigenous fillers silica sand and quartz powder with the conventional silicate fillers, while kaolinite and calcite have shown their equality with kaolin and Winnofil S respectively.

By effecting slight improvements in the manufacturing methods to reduce their particle sizes the two fillers kaolinite and calcite could be made into quite economical and versatile substitutes for imported kaolin and calcium carbonate based fillers.

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