

EFFECT OF EGG SHELL AND RICE HUSK ON HARDNESS AND SWOLLEN PROPERTIES OF NATURAL RUBBER VULCANIZATES .

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ABSTRACT

Egg shell and rice husk were incorporated into natural rubber using a laboratory size two roll mixing mill. A conventional vulcanization was used for curing. The equilibrium swelling test in toluene and hardness characteristics were measured as functions of filler loading. The composites were varied from 0pph to 70pph. The equilibrium swollen of the egg filled vulcanizates exhibited a regular decrease with increase in fillers 290.7 to 196.2 increase in filler and thereafter increased to 254.8, decrease to 195.1 and finally decrease 170.5. That of rice husk decreased from 326.8 to 262.2 and finally increased to 420.7. the hybrid was erratic with decrease from 336.8 to 322.0 and then to 317.6 and finally increased to 341.4. as for the hardness test, both egg shall and rice husk increased with increased in filler contents from 0pph to 70pph.

Keywords: Natural rubber, eggs hall, rice husk, composites, vulcanizate.

INTRODUCTION

Natural rubber (NR) is one of the main elastomers and widely used to prepare many rubber compounding products[1]. Natural rubber is a renewable agricultural resource that does not naturally possess the necessary hardness and modulus required for its commercial acceptability[2]. Natural fillers is frequently reinforced by assimilation of fillers to improve its mechanical properties like: tensile strength, modules, tear strength elongation at break, hardness, compression set, rebound resilience and abrasion resistance [3]. The incorporation of various materials (additives as compounding ingredients) increases these aid characteristics to the level desired for NRs demands [4]. Filler is one of the major additives used in natural rubber compound and has marked effect and influence on rubber materials. Filler play a dominant role in modifying the physical properties of base polymer [5]. In rubber industry, fillers that are commonly in use are carbon black, China clay and calcium carbonate. Nowadays, there has been a growing interest in the use of individual and Agricultural waste such as product like rice husk [6] as fillers for rubber and their blend Rice husk is a major by-product obtained from the production of rice. The main constituents of rice husk are cellulose, lignin and sugar. In addition to organic compounds rice husk is also composed of approximately 20 wt% of amorphous silica by weight of burned pelt [7]. It is the most important agricultural dregs and well recognized that the rice husk is a significant source of silica. [8] worked on an investigation on the potential of Palm Kernel Husk as filler in Rubber Reinforcement. One of the most important phenomena on material science is the reinforcement of rubber by rigid entities, such as carbon black, clays silicates and calcium carbonate [9]. Thus, fillers such as egg shell or reinforcement aids are added to rubber formulations to optimize properties that meet a given service application or set of performance parameters composites. The preliminary results show that palm Kernel Husk is potential reinforcing filler for natural rubber compounds. [10] in analysis of variance of effect of rice husk ash and commercial fillers in NR compounds. The analysis reveals that BRHA shows little variation in the mechanical properties of NR compounds; in other words, it is non-reinforcing

filler and their use must be restricted to 20pht, approximately. For WRHA, NR compositions containing 10 and phr of filler shows a real increment in the tensile strength and the variation of this filler (0 up to 50phr) causes maximum variation upon tensile strength, according to FO value. [11], worked on effect of coconut fibre filler on the cure of characteristics and physic – mechanical and swelling properties of natural rubber vulcanisates. The result shows that coconut fibre is a potential reinforcing filler for natural rubber compounds. Hardness of filled vulcanizate with coconut fibre increased in filler loading. The resistance to swelling of natural rubber compound is dependent on the amount of filler loading; the higher of filler content the lower the equilibrium sorption values obtained.

The mechanical properties of Natural rubber with flyash were investigated and compared with carbonate [12]. From the result, it was observed that the flyash filled composites were better in mechanical properties compared to those filled with calcium carbonate.

This research deals with the effect of egg shell and rice husk on the hardness and swollen properties of natural rubber vulcanizates.

MATERIAL AND METHOD.

Materials

Natural rubber crumbs (Grade NR -10) were obtained from the Rubber Research Institute of Nigeria (RRIN), Iyanomo – Benin. The rubber compounding chemicals such as Zinc oxide, steric acid, Sulphur and CBS were of commercial grade.

Egg shell and rice husks were obtained from Kano metropoly and Danbatta LGA of Kano.

FILLER PREPARATION

Large quantity of both eggshell and rice husk were obtained, cleaned, dried and ground into fine powder; sieved with a mesh of the sizes 120um, respectively.

DRY ASHING

1.0g of each samples of egg shell and rice husk were ashes in a furnace, at 450oc for about 8 hours.

DIGESTION

Eggshell was digested with 4 mol of HNO₃ and made up with deionized water to 100cm³ flasks, filtered into a Labeled sample bottle.

ANALYSIS OF DIGESTED SAMPLES

The samples were analyzed for the element potassium (k), calcium (Ca), sodium (Na), copper (Cu), Zinc (Zn) Mangane (Mn) Magnsium (Mg), lead (pb) and Iron (Fe) with A.A.S.

COMPOUNDING

The recipient used in compounding of the natural rubber compound

Is given in Table 1

Table 1: Recipient for compounding the NR mixture

Ingredient	Grade	Part per hundred (pph)	Part per batch (g)
Natural rubber	NR-10	100	400
Steric acid	Gen. Purpose	1.5	6
Zinc Oxide	Industrial	5.0	20
C.B.S	Industrial	0.6	24
Sulphur	Industrial	2.5	10

C.B.S. = Cyclohexyl Benthlexyl Sulpheamide

CURING

The gum stock of mixture from each formulation was pressed into sheet at the pressure 165kg / cm³ and cured at 155⁰c.

Hardness Test

The hardness of the vulcanizate was determined with Durometer - shore

A, Hardness tester instrument model DIN 53505, in accordance with ASTM D- 2240,

Swollen Test

The swelling property were determined by immersing specified weight of cured sample in toluene solvent in an air tight container, at an ambient temperature, for 24 hours dried and weighed in accordance with ASTM – D3010.

$$\% \text{ Swelling} = \frac{w_2 - w_1}{W_1} \times 100$$

DENSITY

The relative density of vulcanizate composites were determined using specific gravity balance (SG).

The density of rice husk filler was determined by achimedis principles while that of eggshell filler with gravitmetric method, using density bottle.

$$Rs = \frac{W_1 - W_0}{(W_4 - w_0) - (w_2 - w_1)}$$

Where W₁-W₀ = Weight of the filler

(W₄ - w₀)- (w₂ - w₁) = weigh of Volume of water

PH Measurement

This was determined using digital computer pH meter (Luttron 210).

RESULTS AND DISCUSION

Particle size

The particles size of 120um of filler of egg shell and rice husk was used, respectively. Fine particles actually affect their greater interaction between the rubber matrix and the filler and hence provide a higher degree of reinforcement than the coarse ones, in accordance with Hepturn (1984), Fetterman (1985) and a patterman (1976).

Elementary Analysis

It was observed from the result obtained in Table 4.1, that the egg shell has a higher concentration of the elements than that or rice higher, except for Ca Mn and Fe. Therefore, the higher density observed in egg shell us most likely due to the elements that make it up and the degree to which the egg shell particles are bound together in aggregate or agglomeration due to Vander Walls forces, according to Byer (1987)

Table 2: Elemental analysis of egg shell and rice husk.

SAMPLE	ELEMENT (CONC.Mg/L)								
	K	Ca	Ng	Cu	An	Mn	Mg	pb	Fe
Eggshell	23.12	32	4.250	0.050	11.23	0.016	92.13	0.243	0.041
Rice Husk	31.12	0.5	2. 023	0.024	10.62	0.055	91.23	0187	0.803

pH of filler.

The pH results obtained were 8.14 (basic) for egg shell and 5.50 (acidic) for rice husk. Alkaline substance (eggshell) increase the cure rate i.e the chemical cross linking of individual polymer s stated by Stephen (1987).

Filler density

The densities for egg shell and rice husk were 2.313g/cm^3 and 0.833g/cm^3 respectively. This implies that for a given part per hundred (pph) of filler in the matrix, rice husk particles numerical strength will almost be thrice that of egg shell by magnitude present in the ratio 1:3 in density.

SWOLLEN TEST

The equilibrium swelling of the egg shell, rice husk and hybrid filler NR vulcanizate is as shown in table The apparent swollen index for the egg shell and rice husk decreased as the filler loading increased. The rice husk has a lower swollen value compared to that of egg shell, due to its excessive cross – Link, a favorable effect of resistance to swelling, according to [13]. The eggshell shows to be a better filler because the vulcanizate with higher loading was found to be more resistance. This signifies a well cross-linked material, in accordance to [14], that as filler loading increases in rubber matrix, more and more obstacles are created to the diffusing molecules and thus reduce the amount of the penetrated. Equilibrium sorption in organic solvent of affected by the level of cross-link, filler dispersal, nature of solvent and fillers.

Table 3: Swelling of egg shell, rice husk and hybrid composites in Toluene

After 24 hours.

Egg shell		Rice-Husk		Hybrid	
Formulation	Mean wt	Formulation	Mean wt	Formulation	Mean wt
E ₁₀	290.7	R ₁₀	326.8	E ₁₀ R ₅₀	336.8
E ₂₀	244.9	R ₂₀	329.3	E ₂₀ R ₄₀	322.0
E ₃₀	229.6	R ₃₀	279.0	E ₃₀ R ₃₀	338.1
E ₄₀	196.2	R ₄₀	268.6	E ₄₀ R ₂₀	317.6
E ₅₀	254.8	R ₅₀	271.3	E ₅₀ R ₁₀	341.4
E ₆₀	195.1	R ₆₀	267.2		
E ₇₀	170.5	R ₀	420.7		

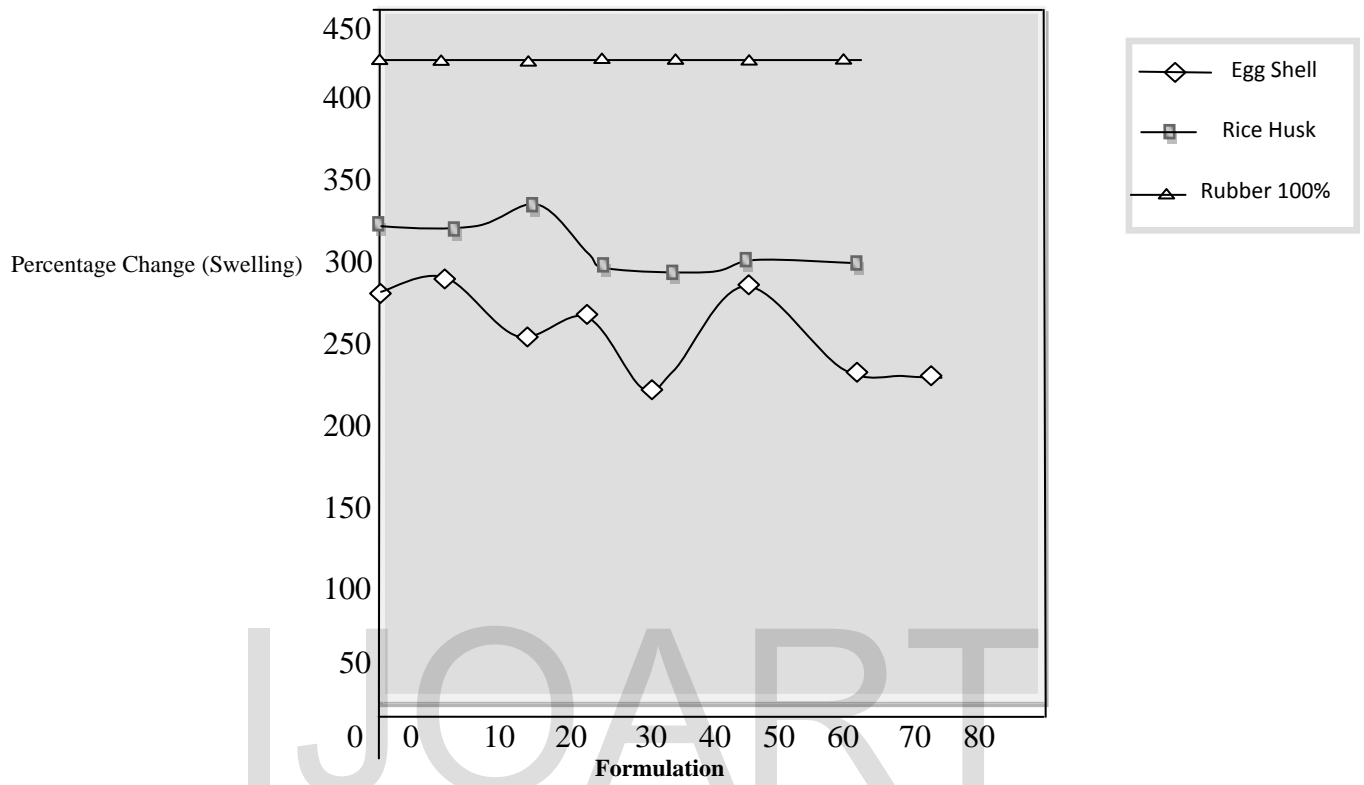


Fig 1a: Effect of formulation on swelling of egg shell and rice Husk Composites in Toluene

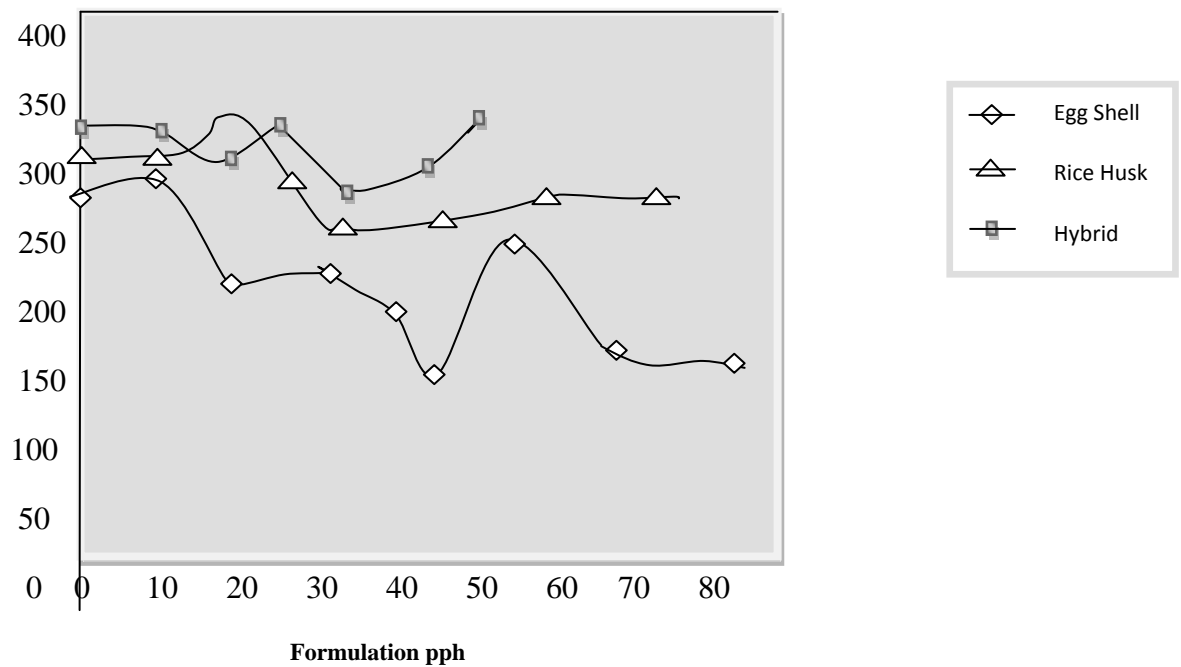


Fig. 1b: Effect of formulation on swelling of Hybrid Composites in Toluene

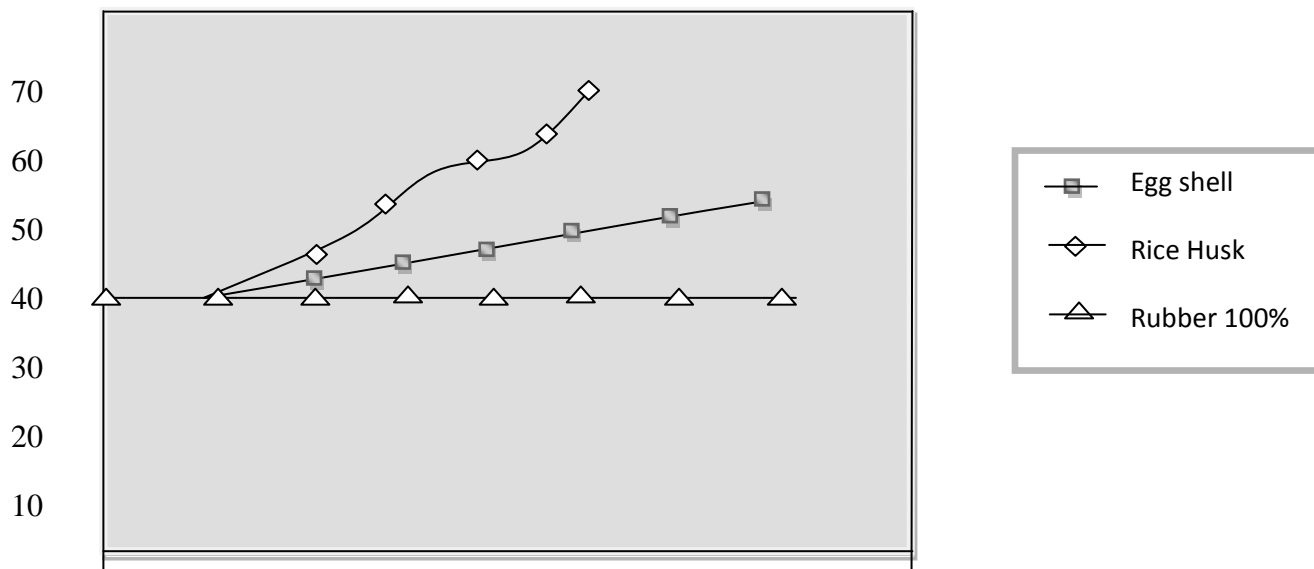
HARDNESS TEST

The hardness result of eggshell, rice husk and the hybrid filled NR vulcanizates is as shown in table 4. The hardness increased with increases in filler content. It was expected because as more filler particles get into the rubber, the elasticity of the rubber chain is reduced, resulting in more rigid vulcanizates in accordance with [15] and [16].

The hardness of rice husk was superior to that of egg shell, due to the difference in their properties, especially the density. This made the dispersion of the rice husk in the rubber matrix more uniform. A poor dispersion reduces filler rubber interaction and consequently decrease the ability of these fillers to restrain gross deformation of the rubber matrix as stated by [17].

Table 4: Hardness test of egg shell, rice husk and hybrid

Egg shell		Rice-Husk		Hybrid	
Formulation (pvh)	Hardness	Formulation (pvh)	Hardness	Formulation (pvh)	Hardness
E ₁₀	40	R ₁₀	40	E ₁₀ R ₅₀	40
E ₂₀	41	R ₂₀	43	E ₂₀ R ₄₀	41
E ₃₀	42	R ₃₀	46	E ₃₀ R ₃₀	42
E ₄₀	43	R ₄₀	50	E ₄₀ R ₂₀	45
E ₅₀	44	R ₅₀	52	E ₅₀ R ₁₀	48
E ₆₀	45	R ₆₀	61		
E ₇₀	47	R ₀	40		



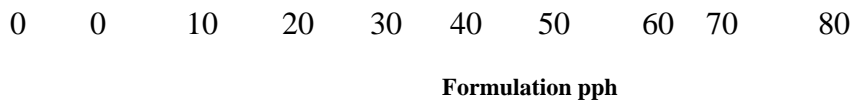


Fig. 2a: Effect of Formulation on Hardness of Egg Shell and Rice Husk Composites

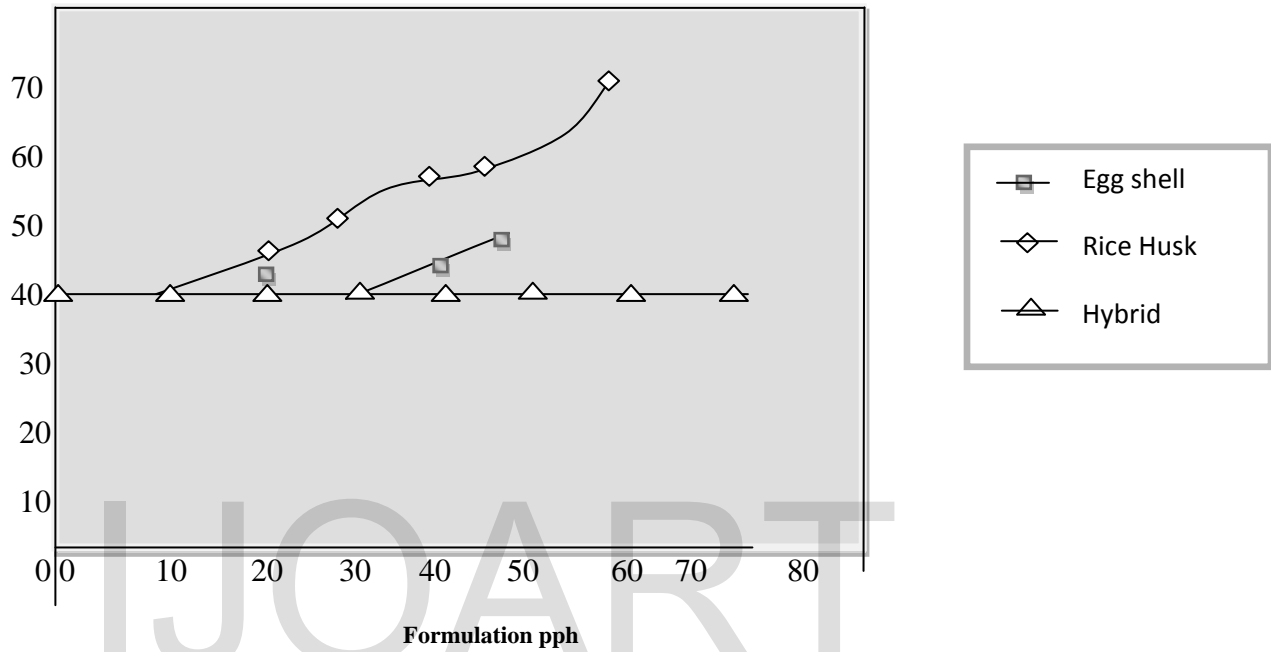


Fig. 2b: Effect of Formulation on Hardness of Hybrid

CONCLUSION AND RECOMMENDATION

The effect of NR and filler – filler interaction on rubber reinforcement was investigated by the use of egg shell and rice husk and the hybrid composites as filler, respectively. The result shows that both fillers were of low reinforcing filler for Natural rubber compounds. However, the eggshell composites exhibited a relatively better reinforcing properties that the rice husk composites. As for the egg shell and rice husk, the analysis revealed that the eggshell showed a little variation better than that of rice husk in hardness swollen properties. The usage must be restricted to 10PPh and 20PPh appropriately, which shows an appreciable increase especially for the variation of the egg shell and rice husk filler can be exploited further by controlling the parties size and distribution, in order to improve the filler dispersion and also its surface functionality.

The result shows that egg shell and rice husk are to some extent are potential reinforcing fillers for natural rubber compounds.

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