



## Physical and Mechanical Properties of Starch/Natural Rubber Biopolymer Blends

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### Abstract

The effects of starch fillers on the physical and mechanical properties of starch/natural rubber blends were investigated with reference to carbon black filled natural rubber. Starch was extracted from three plant crops namely, amora, potato and yam and applied as fillers in natural rubber formulations. Starch/natural rubber (NR) blends were prepared by directly mixing and co-coagulating rubber latex and gelatinized starch by a coagulating agent via latex compounding method. There was improvement in tensile strength and elongation at break especially at 30 phr filler loading for all the starch/natural rubber blends in comparison with the carbon black filled natural rubber. Although the carbon black filled natural rubber at 30 phr gave the highest tensile strength (87.6 Mpa), it however produced a moderately low elongation at break of 6807 compared with amora starch filled NR (10007), potato starch filled NR (6654) and yam starch filled NR (12440) respectively. Carbon black filled natural rubber produced the highest value in hardness (66-68) compared with the starch filled rubber samples at 30 phr filler loading. It was further observed that filler loading restricted swelling in toluene solvent probably as a result of filler – rubber interaction and adhesion and this phenomenon increased with filler loading from 10 phr to 30 phr in the starch/natural rubber blend and also in the reference sample. However filler loading from 10 phr to 30 phr in all the samples increased the degree of crosslink density ( $V_c$  mol cm<sup>-3</sup>).

### Keywords

Starch; Natural rubber; Amora; Potato; Yam; Filler; Carbon black

### Introduction

*Hevea brasiliensis* (rubber tree) has been the source of natural rubber (cis 1,4-polyisoprene) for many years. Natural rubber (NR) is usually harvested from the milky white latex from the rubber tree [1]. Uncured natural rubber (non-vulcanized) has little or no mechanical properties of interest in tyre tread manufacture because of its poor tensile strength and viscoelastic properties.

Sulphur is the principal curing agent in natural rubber in addition to other compounding and curing additives. However, the usefulness and application of cured natural rubber in terms of tensile strength, tear strength, hardness and abrasion resistance will not be complete without the incorporation of carbon black as a reinforcing filler [2-4].

For many years, carbon black has been the most widely used reinforcing filler in natural rubber compounding especially in automobile tyre manufacture [4].

Furnace black type of carbon black is the most popular type in rubber reinforcing fillers in contemporary usage because of its fine particle size, and the properties it confers on the rubber products. More than 90% of the carbon black produced is used by the rubber industry as a reinforcing filler in tyres, tubes, conveyer belts, cables, rubber profiles and other rubber goods [2]. Carbon black is widely known to increase the tensile strength, abrasion resistance, hardness and tear strength in rubber based articles, and also increases the strength of elastomers (both natural and synthetic rubbers) by up to 10 to twenty times [2]. However, owing to carbon black origin from petroleum, its use in rubber compounds not only consumes much energy, but it is also harmful to our environment and its fine particles at prolonged exposure cause easy lung damage and occupational illness for rubber compounding operator and employees in the rubber processing industries [5,6]. More so, the energy crises of the 1970s and 1980s and the high price of crude oil and natural gas was an enough incentive to seek naturally occurring substitutes (as fillers) in rubber polymers [7].

Therefore, it is essential and expedient to develop new cheap, clean, renewable, biodegradable and environmental friendly reinforcing filler for rubber products [5]. The earliest research efforts in incorporating starch into rubber matrix began with in the 1970s where they prepared crosslinked starch xanthide-rubber master batches by co-precipitating mixtures of soluble starch xanthide and elastomer lattices during which starch xanthide was crosslinked. In prepared curds, starch is the continuous phase and the latex particles as the disperse phase. The starch-elastomer crumb was further extruded to produce a phase inversion and as a consequence, it was demonstrated that cross linked starch xanthide could be used as an effective rubber reinforcing agent especially in improving the fatigue life and abrasion resistance, when used to partially replace carbon black in a premium tread in automobile tyres.

More recently, in the patent literature [8], starch has been creatively used as a filler in tyre compounds in order to lower rolling resistance, tyre weight and carbon (iv) oxide emission when used to partially replace carbon black and silica in automobile tyres. Goodyear Tyre co., for example used a new starch based material called BioTRED, in its automobile tyre production and the Goodyear GT3 was claimed to be the first tire on the market using BioTRED. However the starch used in this new development are specially modified corn starch in terms of amylose/amylopectin ratio. Various research efforts has been reported on blending modified natural rubber and functionalized starch with various chemical agents ranging from maleic anhydride, dimethyl aminoethyl methacrylate (DMAEMA) in rubber and resorcinol-formaldehyde (RF) and silane compound (KH792) and various percentage of glycerol or sorbitol in gelatinized starch to obtain various rubber blends of diverse properties and structures [9,10].

Starch, a semi-crystalline biopolymer stored in granules as a reserve in most plants is renewable, non-toxic and completely biodegradable [11,12]. It is also one of the substances more widely

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distributed in nature and consist of amylose and amylopectin in considerable amounts [10]. Both granular and deconstructed starches have been used in combination with other polymers such as poly (propylene carbonate), Poly caprolactone, poly (butylene succinate), poly (3-hydroxy butyrate, poly (lactic acid), and poly (vinylalcohol) [13-15].

The thermoplastic properties of starch have been studied extensively and are directly related to presence of water, glycerol or sorbitol [16]. More so, in its dry state, the glass transition temperature (T<sub>g</sub>) of starch is above its degradation temperature [9]. Furthermore, the mechanical properties of starch-filled rubbers actually depend on many factors among which the most important ones are the dispersion level, the interfacial adhesion/strength, and the type of starch. Generally, it is found that the reinforcing action of a filler depends upon its nature (chemical composition), particle size, type of elastomer and the amount of filler present (optimum quantity), [3]. It has also been reported that amylose content of starch can affect mechanical properties in starch/rubber blends [9]. Amylose also plays a big role in the pasting properties of starch thus affecting the starch viscoelastic properties [17], and in addition, an increase in amylose content of starch has been found to lower swelling power and solubility of starches.

However, the polar surface, high melting point, and large particle size of starch make it difficult to finely disperse starch in rubber matrix. Therefore, incorporation of starch into rubber by direct blending (melt-mixing) has resulted in a micro-composite with large, dispersed particles, which caused significant deterioration of the mechanical properties of vulcanizates, especially the resistance to abrasion and the tear properties. Applying a latex compounding method using gelatinized starch helps to eliminate the above mentioned problems to an appreciable extent. In this research effort, starch was extracted from plant source other than corn (maize) from amora, sweet potato and yam and were applied as fillers in an unmodified state (natural and unfunctionalized) but in a gelatinized form reducing particle size and dissociating the hydrogen bond in starch; thereby paving way for rubber latex particles which are generally smaller than 100 nm to be mixed with starch paste uniformly and directly co-coagulated with a coagulating agent to give a blend with a fine dispersion. The effects of the above mentioned starch fillers (amora, sweet potato and yam) on the rubber blends were investigated with reference to a carbon black filled rubber in terms of tensile strength, hardness, elongation at break, crosslink density and swelling properties in a solvent.

The investigation into the relationship between microstructure, thermal stability, chemical composition, and mechanical properties of the starch/natural rubber blends are ongoing at various levels and the results are being gathered and collated appropriately.

## Methodology

### Materials

Local variety of the starchy crop, amora (Polynesian arrow root), *Tacca leontopetaloides* was harvested from a cassava farm in Paiko village, Gwagwalada Area Council, FCT, Abuja, Nigeria while sweet potato, *Ipomoea batatas* and yam tuber, *Dioscorea rotundata* were purchased from food market in Mubi, Adamawa State and Ramat park food market, Benin city, Edo state, Nigeria.

Natural rubber latex (dry rubber content 30%) was obtained from Rubber Research Institute of Nigeria (RRIN) Iyanomo, Edo State, Nigeria. The chemicals, Calcium Chloride and toluene were

purchased from chemical dealers in Benin and Abuja respectively. Compounding chemical additives were also purchased from chemical dealers in Benin city, Edo State, Nigeria.

### Preparation of starch/natural rubber blends

Starch was extracted from the crops, amora, sweet potato and yam tuber was characterised in terms of particle size, pH and amylose and amylopectin percentage composition as reported [18]. The gelatinized starch was prepared according to the method of Zhang et al. (2004) with starch content in parts per hundred rubber (Phr) of 10 phr, 20 phr and 30 Phr respectively for amora, sweet potato and yam starch. The starch aqueous suspension was stirred at 90°C in a water bath for 30 minutes until the solution became transparent. When the solution cooled to ambient temperature, a starch paste (gelatinized starch) was obtained. The gelatinized starch and the rubber latex (30% DRC) were mixed and stirred vigorously for 30 minutes to harmonize properly and then 10% wt/wt Calcium Chloride aqueous solution was added to co-coagulate the rubber latex and gelatinized starch. The coagulum were washed in water and passed through the rollers in the Lohashilpi sheeting machine and compressed into thin sheets with embossed ribbed pattern. The coagulum sheets were washed in water and allowed to drain off water droplets and then cut into thin shreds (short strands) with scissors, labelled and placed in a dry oven at 80°C for 18 hours and prepared for compounding and vulcanization.

### Compounding procedure

The formulation for the compounding of the starch/natural rubber blend is shown in Table 1. The compounding was carried out in accordance with American society for testing and materials (ASTM) D3184-80 in a two roll mill and was cured using sulphur as curing agent, according to the formulation in Table 1. The control sample (reference) Ro, was also compounded and filled with carbon black at 30 Phr. Test samples of dumbbell shapes (3 pieces) each were punched out from the vulcanized starch/rubber sheets for mechanical testing on Instron Universal Testing Machine (IUTM).

**Key:** MBT: Mercapto Benzothiazole (Captax); TMQ: Tetra Methyl Quinolone

### Characterization of the starch/natural rubber blends

Tensile strength (Mpa) and elongation at break (%) were determined using a Lloyd instrument machine, Instron universal testing machine, according to ASTM D412 at a speed rate of 500 mm/min using three (3) dumbbell test pieces.

### Hardness test

Type A durometer gauge was used to measure hardness in the starch/rubber vulcanizates according to ASTM D2240.

**Table 1:** Shows typical formulation for the starch/rubber blends in part per hundred rubber (Phr).

Components	Quantity (Phr)
Natural rubber	100
Starch	10, 20, 30
Zinc oxide	5
Stearic acid	2
MBT	3
TMQ	1
Sulphur	3
Carbon black	30

## Swelling tests

Swelling tests on the starch/natural rubber vulcanizates were carried out in toluene solvent according to ISO 1817. The cured test pieces (round shape pieces) were weighed accordingly and swollen in toluene until equilibrium is reached (72 hours) at room temperature as indicated by constant weight. The starch/rubber samples were taken out from the toluene, cleansed with tissue paper and reweighed using a weighing balance. The samples were then dried in the oven at 60°C for 2 hours until constant weight is obtained. The swelling tests results were used to calculate the molecular weight between two crosslink (Mc) and the degree of crosslinks density (Vc) applying Flory Rehner equation:

$$Mc = \frac{-pp \ V_s \ V_r^{1/3}}{\ln(1 - V_r) + V_r + \chi V_r^2} \quad (1)$$

$$V_r = \frac{1}{1 + Qm} \quad (2)$$

$$V_c = \frac{1}{2Mc} \quad (3)$$

Where p, is the rubber density (p of NR=0.92 g/cm<sup>3</sup>) V<sub>s</sub> is the molar volume of the toluene (V<sub>s</sub>=106.4 cm<sup>3</sup>/mol), V<sub>r</sub> is the volume fraction of the polymer in the swollen specimen, Qm is the weight increase of the blends in toluene and χ is the interaction parameters of the rubber network-solvent (χ of NR=0.393). Other swelling parameter calculated include the mol% uptake of the solvent (Qt), swelling index percentage and the swelling coefficient,

$$\frac{w_2 - w_1}{w_1} \times 100 \quad (i)$$

$$\text{Swelling index\%} = \frac{w_2 - w_1}{w_1} \times 100 \quad (ii)$$

$$\text{Swelling coefficient} = \left[ \frac{A_s}{m} \right] \times \left[ \frac{1}{d} \right]$$

Where W<sub>1</sub>=initial weight of polymer before swelling

W<sub>2</sub>=final weight of polymer after swelling

M<sub>s</sub>=molar mass of the solvent

A<sub>s</sub>=weight of the solvent sorbed at equilibrium swelling

m=mass of the sample before swelling

d=density of the solvent used

## Results and Discussion

**Key:** Ro=Natural rubber with carbon black filler (control)

Phr=Part per hundred rubber

IRHD=International Rubber Hardness Degree

**Key:** Ro=Natural rubber with carbon black filler (control)

Phr=Part per hundred rubber

A=Amora

**Key:** Ro=Natural rubber with carbon black filler (control)

Phr=Part per hundred rubber

P=Sweet Potato

**Key:** Ro=Natural rubber with carbon black filler (control)

Phr=Part per hundred rubber

Y=Yam

## Mechanical properties of starch/natural rubber blends

The mechanical properties of starch/natural rubber blends with starch fillers and carbon black are presented in Tables 2-5 for amora, sweet potato and yam respectively. There was a decrease in tensile strength, elongation at break and hardness as the filler loading increased from 10 phr to 20 phr in terms of tensile strength and elongation at break. Hardness decreased continuously with increase in starch content. However the control rubber blend filled with carbon black, maintained a lead in tensile strength and hardness but a moderately low elongation at break at 6807. The finer particle size of carbon black (10-50 nm), spherical shape, concentric graphitic layers of carbon and the wetting by the rubber polymer must have contributed to the high tensile strength and hardness of the natural rubber blend filled with carbon black [19]. At the same time, it can be seen that at 30 phr filler loading with carbon black, there was a decrease in elongation at break meaning that resilience decreased at 30 phr loading. It can be inferred that carbon black restrict the mobility and deformability of the matrix macromolecules at higher degree with increased carbon black loading.

From Table 4 the tensile strength and elongation at break increased from 10 phr to 30 phr for potato starch filled rubber but decreased at 30 phr for yam starch filled rubber. Whereas the hardness (58-59) in potato starch filled rubber increased at 20 phr loading but came down to 52-53 at 30 phr, the hardness in yam starch filled rubber increased with increase in the filler content of up to 30 phr consistently till 30 phr.

**Table 2:** Particles size, pH, amylose and amylopectin composition of the starch samples.

Components	Particles size (μm)	PH	Amylose %	Amylopectin%
Amora	156 × 47	6.9	14.86	84.97
Sweet potato	153 × 46	5.2	22.34	77.64
Yam	155 × 40	6.8	29.68	70.29

**Table 3:** Effect of amora starch on mechanical properties of natural rubber blends.

Filler quantity (Phr)	Tensile strength (MPa)	Enlongation of break (%)	Hardness (IRHD)
10	13.3	4850	56-57
20	8.9	2883	52-53
30	57.4	10007	47-48
Ro	87.6	6807	66-68

**Table 4:** Effect of sweet potato starch on mechanical properties of natural rubber blends.

Filler quantity (Phr)	Tensile strength (MPa)	Enlongation of break (%)	Hardness (IRHD)
10	11.5	3137	54-55
20	12.9	3490	58-59
30	35.7	6654	52-53
Ro	87.6	6807	66-68

**Table 5:** Effect of yam starch on the mechanical properties of natural rubber blends.

Filler quantity (Phr)	Tensile strength (MPa)	Enlongation of break (%)	Hardness (IRHD)
10	9.5	2544	54-55
20	76.1	11274	57-58
30	32.3	7588	60-61
Ro	87.6	6807	66-68

**Table 6:** Effect of amora starch on the swelling properties of natural rubber blends.

Filler quantity (Phr)	Initial weight (g) (W1)	Final weight (g) (W2)	Solvent sorbed at swelling (W2-W1)	Mol % uptake Qt	Swelling index (%)	Swelling coefficient	Degree of crosslink density (vc(10 <sup>-5</sup> ))	Molecular weight between two cross link (Mc)
A10	1.80	7.50	5.70	3.44	317	3.66	33.5	149.46
A20	1.90	7.70	5.80	3.32	305	3.52	33.1	151.10
A30	1.6580	5.3143	3.6563	2.40	221	2.55	44.0	113.62
Ro	2.0284	4.8023	2.7739	1.49	137	1.58	51.9	96.36

**Table 7:** Effect of sweet potato starch on the swelling properties of natural rubber blends.

Filler quantity (Phr)	Initial weight (g) (W1)	Final weight (g) (W2)	Solvent sorbed at swelling (W2-W1)	Mol % uptake Qt	Swelling index (%)	Swelling coefficient	Degree of crosslink density (vc(10 <sup>-5</sup> ))	Molecular weight between two cross links (Mc)
P10	1.90	6.90	5.0	2.86	263	3.04	36.3 × 10 <sup>-5</sup>	137.69
P20	1.84	7.02	5.18	3.06	282	3.25	35.5 × 10 <sup>-5</sup>	140.82
P30	1.8257	6.5722	4.7465	2.83	260	3.00	37.5 × 10 <sup>-5</sup>	133.34
Ro	2.0284	4.8023	2.7739	1.49	137	1.58	51.9 × 10 <sup>-5</sup>	96.36

**Table 8:** Effect of yam starch on the swelling properties of natural rubber blends.

Filler quantity (Phr)	Initial weight (g) (W1)	Final weight (g) (W2)	Solvent sorbed at swelling (W2-W1)	Mol % uptake Qt	Swelling index (%)	Swelling coefficient	Degree of crosslink density (vc(wo <sup>-5</sup> ))	Molecular weight between two cross link (Mc)
Y <sub>10</sub>	1.80	6.80	5.0	3.02	278	3.2	36.3 × 10 <sup>-5</sup>	137.69
Y <sub>20</sub>	1.83	6.48	4.65	2.76	254	2.93	37.9 × 10 <sup>-5</sup>	131.71
Y <sub>30</sub>	1.7085	5.8060	4.0975	2.61	240	2.77	41.1 × 10 <sup>-5</sup>	121.78
Ro	2.0284	4.8023	2.7739	1.49	137	1.58	51.9 × 10 <sup>-5</sup>	96.36

The carbon black filled rubber still maintained the lead in tensile strength and hardness but moderately low elongation at break indicating that carbon black at 30 phr restricts the mobility and resilience of the rubber blend as previously shown.

According to Tables 6 and 7, the mol % uptake of the solvents, swelling index and swelling coefficient decreases as the filler loading increases from 10 phr to 30 phr in the starch/natural rubber blends, while the degree of crosslink density (Vc) is inversely proportional to the molecular weight between two cross links (Mc) in other words, as the Vc increases the Mc, decreases consistently. In Table 8 for both mol % uptake Qt, swelling index % and swelling coefficient, the values increased at 20 phr and decreased at 30 phr. This behaviour could be explained by the particle size of potato starch which is the biggest particle size among the starch fillers as there is no perfect miscibility among mixed polymer pairs in a blend [3].

## Conclusion

From this study, the following conclusions can be drawn.

1. The increased filler loading with starch from 10 phr to 30 phr affects tensile strength, elongation at break and hardness positively except in few cases where particle size, inadequate wetting of the fillers by the rubber brings some drawback in properties [20-26].
2. The degree of crosslink density, Vc is inversely proportional to the molecular weight between two crosslinks, Mc. More so, filler loading from 10 phr to 30 phr restricts swelling in toluene solvent as a result of the filler rubber interaction and adhesion at the interfaces as evidenced by; increased value of crosslinks density and no phase separation among the rubber composite [20,27-30].

3. Filler loading of starch from 10 phr to 30 phr affects the elongation at break positively in rubber blends and hence better resilience in the blends even more than the carbon black reinforced rubber blend, (the control) though with lesser tensile strength compared to Carbon black filled rubber blend [31-35].

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