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2. Comparison of Reinforcing Fillers used in Natural Rubber Latex Films

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ABSTRACT

A major problem with natural rubber latex used in thin film manufacturing is having less control over the properties of the material and low resistance to heat and ageing, compared to Synthetic rubber products. Since the industries are moving towards environment friendly solutions, in the current context, and high comfortability and durability of the wearable products, it is important to find means to enhance the properties of the NRL products. Introducing fillers into NRL is a common solution used in this cause. Carbon black, silica, calcium carbonate can be identified as widely used traditional fillers. Literature shows that the mechanical response of NRL products reinforced with fillers is highly influenced by several parameters such as, the processing conditions, the homogeneity of filler dispersion, the NR-filler interaction and the Morphological aspects of fillers. With the latest trend in field of science on graphite and its derivatives, several attempts have been carried out on introducing graphene and graphene based materials into natural rubber latex products. This review is a comparison carried out on the performance of Graphene and Graphene based materials used as a filler in NRL films.

Nomenclature:

NRL	natural rubber latex
sp2	The mixing of one s and two p atomic orbitals
CNT	Carbon nanotubes
ISO	International Organization for standardization
ASTM	American Society for testing and materials
TS	Technical Specifications
CVD	Chemical Vapour deposition
SiC	Silicon Carbide
XRD	X-ray Diffraction

UVVis	ultraviolet visible spectroscopy
TEM	transmission electron microscopy
SEM	scanning electron microscopy
Phr	parts per hundred rubber
rGo	Reduced graphene oxide
GO	Graphene oxide
GNP	graphene nano plates
TS	Tensile strength
Sp3	The mixing of one s and three p atomic orbitals.

1. Natural Rubber composites, Graphene and Graphene Based Materials:

Carbon is a versatile element found in the earth and carbon materials generate an abundant group of other materials which have a broad range of applications. Carbon black is a by-product of the petroleum industry, produced by the incomplete combustion of heavy hydrocarbon fuel. Carbon black is widely used as a filler and reinforcement materials in dry rubber products, such as tires and other natural rubber products.

When carbon black applied to NRL films it only acts as a filler material and it does not supply much reinforcement to the film[1]. Graphene and graphene based materials have been incorporated into NRL to produce reinforcement and functional properties[6].

Graphene has been anticipated as a promising novel material for modern years due to its outstanding physical properties such as good electron mobility, good thermal conductivity, strength, mechanical stiffness, high surface area and elasticity. Therefore graphene and graphene based materials act as a versatile and economical filler in NRL even at very low concentrations.

Graphene is a thick sheet of sp2 hybrid carbons covalently bonded to three other carbon atoms with a carbon bond length of 0.142nm, forming a hexagonal ring structure. Graphite, a 3D (3D) layered lattice structure, is formed by stacking two-dimensional (2D) parallel sheets of graphene.

Adjacent graphene sheets of graphite are held together by low van der Waals forces with a separation distance of 0.335 nm. The pz orbitals of the carbon atom can overlap more efficiently when they are parallel; therefore, graphene sheet has the lowest energy when completely flat.

The π orbitals are distributed throughout the graphene sheet, making it both thermally and electrically conductive. Graphene is the basic building block of all other forms of carbon graphite such as nanotubes (CNT) and fullerenes [30].

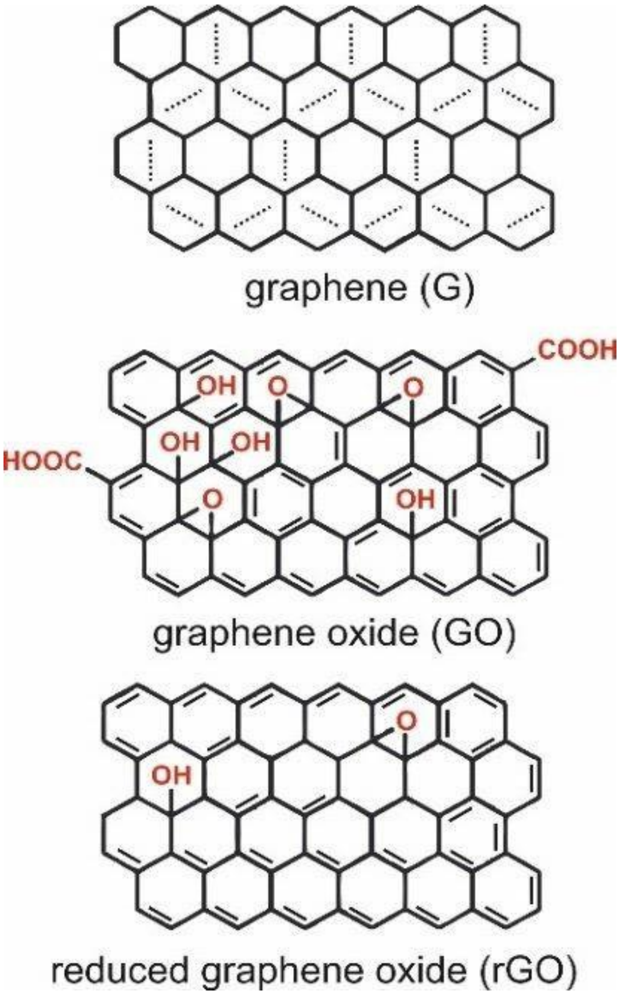


Figure 1.1 Structure of Carbon Atom Arrangement in a) Graphene(G) b) Graphene oxide(GO) c) Reduced

1.1 Graphene as a Filler in NRL Films:

Graphene is a single layer of sp2-hybridized carbon atoms arranged in a two dimensional lattice. Although graphene has been studied in theory for decades, its real existence was not proven until 2004, when Kostya Novoselov and Andrei Geim from the Manchester University managed to isolate a monolayer of graphene from graphite for the first time. In 2010 they were awarded the Nobel Prize[3]. According to the most recent ISO standard (ISO/TS 80004-13:2017) it is a single layer of carbon atoms which bonds covalently with three neighboring carbon atoms in a honeycomb structure[4].

Generally graphene is produced under two methods, 1) top down processes (eg -mechanical milling[3,5], Chemical exfoliation etc.[6] 2) bottom up processes (eg-chemical vapor deposition (CVD)[7], chemical conversion[8,9] epitaxial growth on SiC [10,11], arc discharge [7] etc.

Bottom up processes yield very high quality graphene with very low defects which could be suitable for biomedical and electronic applications etc.

Since Bottom up processes are expensive and not capable of used in large scale, top down approach is more preferable for applications such as sensors, batteries, inks and fillers in composites.

Most common top down processes reported are chemical exfoliation methods such as Hummers method, Liquid phase exfoliation, graphite oxide/fluoride reduction, electrochemical expansion, intercalation compound exfoliation[15] and exfoliation of graphite with Triazine derivatives under ball milling conditions[16].

In the last method 2,4,6-triamino-1,3,5-triazine (Melamine) has performed the best performance as an exfoliation agent. This method could be more suitable for NRL composite preparation because it is easy to perform, inexpensive, environmentally friendly [17] and it also does not adversely affect the colloidal stability of the NRL.

Characterization of graphene is an important part of graphene reinforced NRL production. This involves investigation of graphene structure, number of layers, defects and properties etc.

The most often used characterization techniques are Raman spectroscopy, X-ray diffraction(XRD), ultraviolet visible spectroscopy (UVVis), transmission electron microscopy (TEM), scanning electron microscopy(SEM) and optical microscopy.(31)

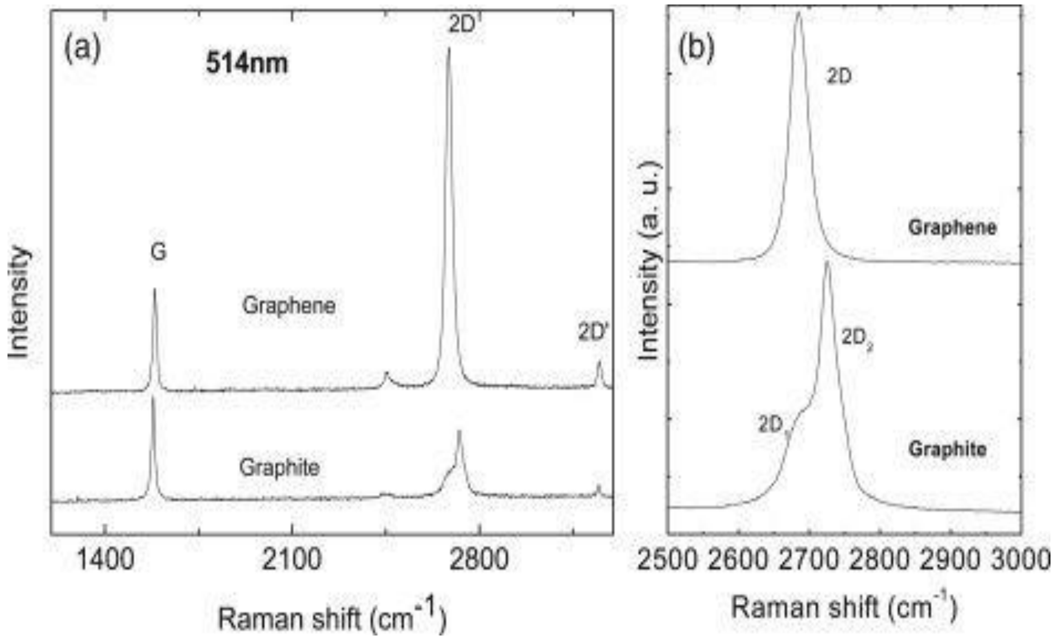


Figure 1.1 1 (a) Comparison of the Raman spectra of graphene and graphite measured at 514.5 nm. (b) comparison of the 2D peak in graphene and graphite[18]

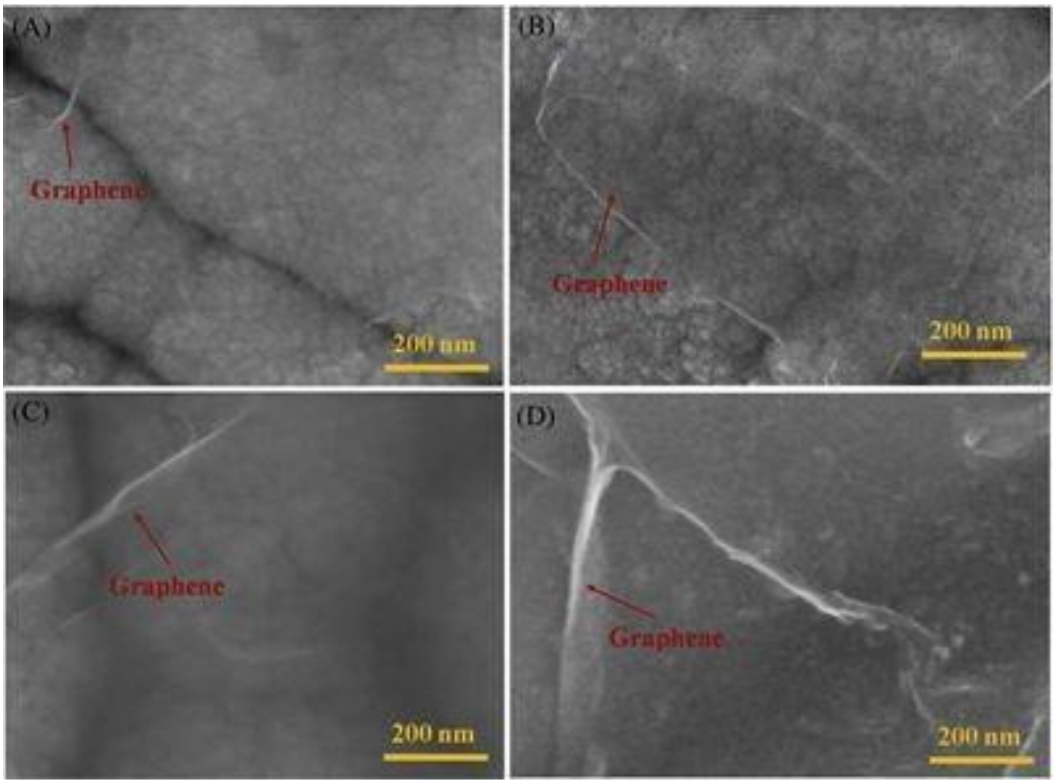


Figure 1.1 2 SEM images of (A) mono layer, (B) bilayer, (C) three layer and (D) multilayer Graphene films on Alumina substrate[19]

It has been found that composite properties vary considerably when the filler particles each a high degree and a high level of homogeneity in terms of dispersion. Among the different factors which affect the dispersion of graphene in NRL is the most important step in fabrication of NRL films.

Dispersion, the type of interaction between the filler and the NRL at the interface has a significant effect on the final properties of the composite.

Therefore efforts to enhance the interface interaction is a prominent feature in each manufacturing techniques. [7]

Generally elastomer composites can be synthesized using three methods, Namely solution mixing (dispersion method), melt intercalation and in situ polymerization. These techniques can be also used in manufacturing of composites of NRL with graphene and graphene based materials.

Different instruments such as ultrasonicators, mechanical stirrers and magnetic stirrers etc are used in latex suspensions for film manufacturing. Depending on the required properties mixing method and the instrumentations can be selected.

Figure 1.1 3 Probe Sonicator

Gejo George-2017[12], observed the properties of graphene incorporated NRL films. In this study Graphene was synthesized using planetary ball milling by exfoliation of graphite with melamine (ratio 1:3). Then the ball milled material was made into 30% aqueous dispersion by probe sonication and incorporated into NRL at different graphene loadings [0.3 (0.29), 0.7 (0.66), 1.5 (1.43), 3 (2.85) and 5 (4.75) phr (wt.%)] by probe sonication again. Dip molding was done to fabricate thin films for tensile, thermal conductivity and electric conductivity analysis the samples were cast on a glass plate surface. Tensile testing on samples were done using the Shimadzu AGX-10 universal testing machine (UTM) and thermal conductivity of graphene-NR latex nanocomposite samples were measured using a HOTDISK TPS 2500S Thermal Constants Analyser under two loadings(200 g and 500 g). Electrical Conductivity of graphene-NRL nanocomposite samples were measured using Hioki 3532-50 LCR Hitester using the two probe method.

The optimum properties were obtained by the 1.5 phr (1.43 wt.%) graphene concentration, which showed 40% increase in tensile strength, (480-980 %) increase in thermal conductivity and 60% improvement in electric conductivity.

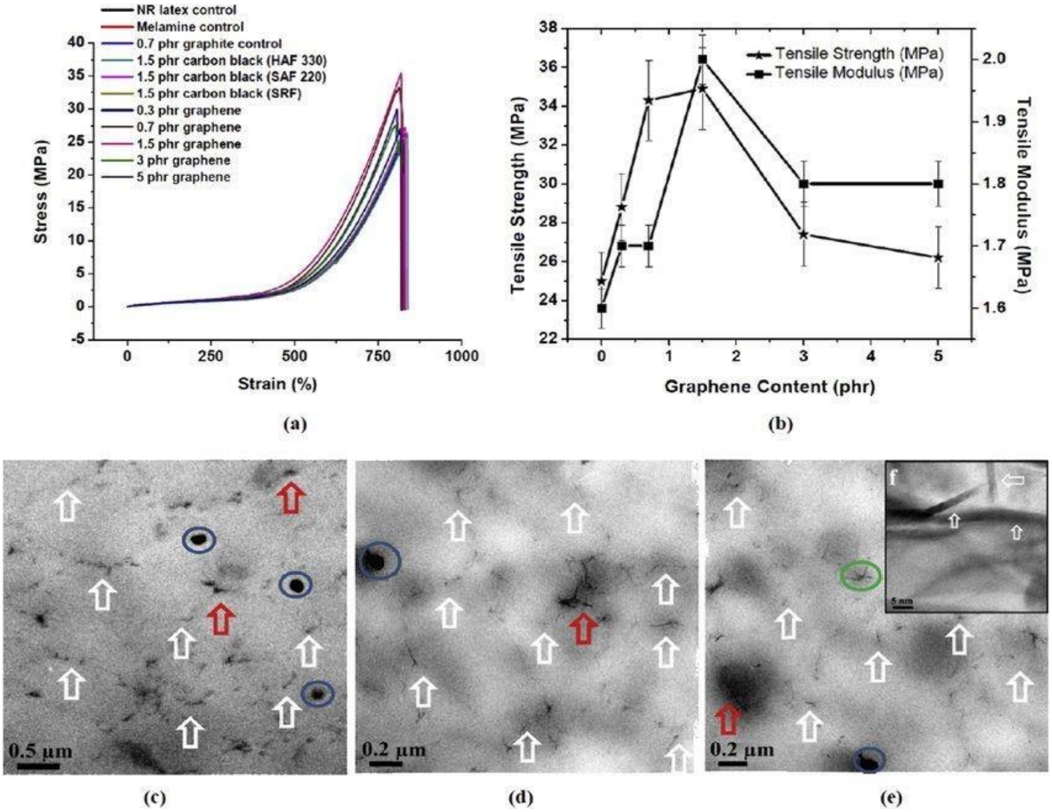


Figure 1.1 4 a) Representative stress-strain curves of all the control samples and graphene-NR latex nanocomposite samples, b) Tensile properties of graphene reinforced NR latex composites, (c), (d) and (e) TEM images of graphene-NR latex nanocomposite thin films (12)

The increment in thermal conductivity in this study is far superior than the previously reported studies on rGo NRL nanocomposites[13,14]. Attaining higher thermal conductivity in graphene-polymer composites is mainly limited by two parameters i) quality of the graphene (defects, isotopes, impurities or vacancies) and ii) the large interfacial thermal resistance between the polymer mediated graphene boundaries.

In order to achieve higher thermal conductivity, By some other researchers have tried higher loading of fillers[20] which affect the physical properties of the composite. This study has established comprehensively the “proof of principle” that well dispersed and stable aqueous dispersions of few layer graphene-NR latex nanocomposites is a desirable precursor for producing new generation of rubber-graphene composites for numerous applications.

Furthermore, they’ve proposed that this concept can be adopted to produce a range of defect free few layer graphene-synthetic latexes/emulsions/polymer dispersions-based nanocomposites to significantly increase its physical, mechanical, thermal and electrical properties.

1.2 Graphene Oxide as a Filler:

Graphene oxide is a chemically modified graphene which retains much of the properties of pure graphene. It is typically synthesized by oxidation and exfoliation of graphite bearing oxygen containing functional groups, such as hydroxyl (-OH), epoxy (-O) or carboxyl (-OOH) groups. (Figure 1) on their base planes and edges which tend to attractively grow chemical structures at its surfaces.

These groups are strongly hydrophilic which allow graphene oxide to have good contact with functional groups on the surface of Natural rubber latex.

The most reliable and thoroughly used method of preparing GO is the modified Hummers' method[21,22].

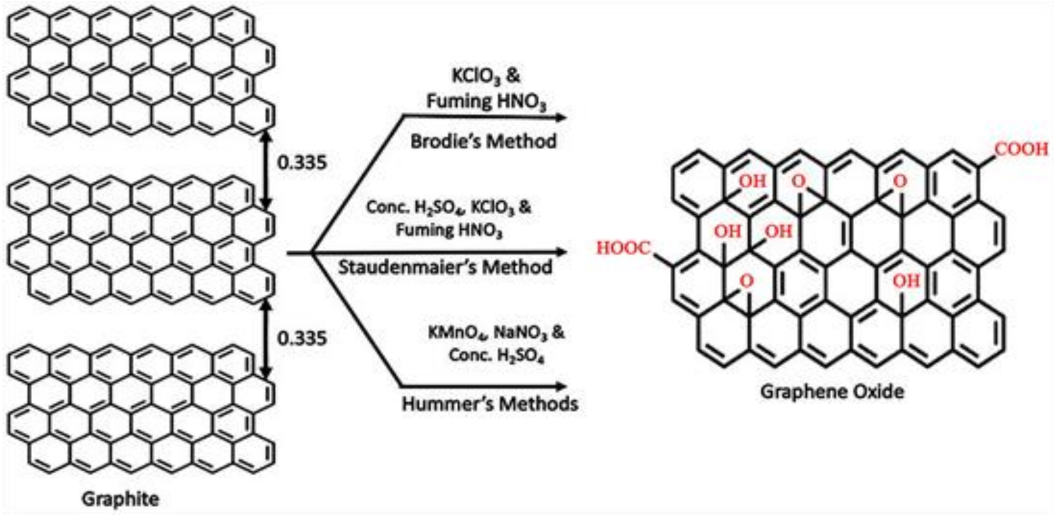


Figure 1.2 1 GO synthesis using a) Brodie's method b) Staudenmaier's method c) Hummer's Method

Kai-Ying Chong[23] and Tutchawan Siriyong[24] observed the properties of graphene oxide incorporated NRL films.

In Kai-Ying Chong's (2018) study GO was synthesized using the modified Hummers' method and then it was neutralized by washing with deionized water.

Low ammonia pre vulcanized NRL was incorporated with GO by solution mixing. NRL and GO were slow speed mechanically stirred and films were formed by coagulant dipping method.

The puncture resistance was measured on a universal testing machine according to ASTM F1342. A puncture needle probe A (round tip: R 0.010 ± 0.001″, length 2.00 ± 0.062″, diameter 0.080 ± 0.002″)

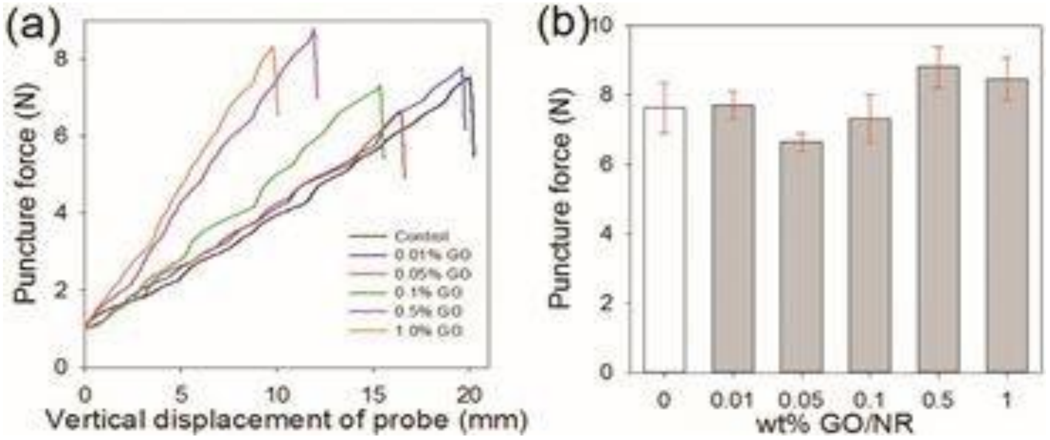


Figure 1.2 2 cture resistance of GO/NRL under different loadings[23]

According to the test results under different loadings, the addition of 0.5 and 1.0 wt. % graphene oxide has considerably increased the puncture resistance of GO/ NR latex films, almost 12-15% increase compared to control sample.

In Tutchawan Siriyong study in 2014,[24] GO was synthesized using the modified Hummers' method and then it was neutralized by washing with deionized water.

GO filler dispersion was prepared by bath sonication, then pre vulcanized NRL was incorporated with GO by ultra sonication.

Lastly NRL films were produced by coagulant dipping method and air vulcanized at 120°C for 15 minutes.

Tensile properties of samples were measured using Instron tensile tester according to ISO 37:2005 and sheet resistance were measured using a PMR 001 4-point probe resist meter, where both contact resistance were eliminated and the bulk resistivity was obtained.

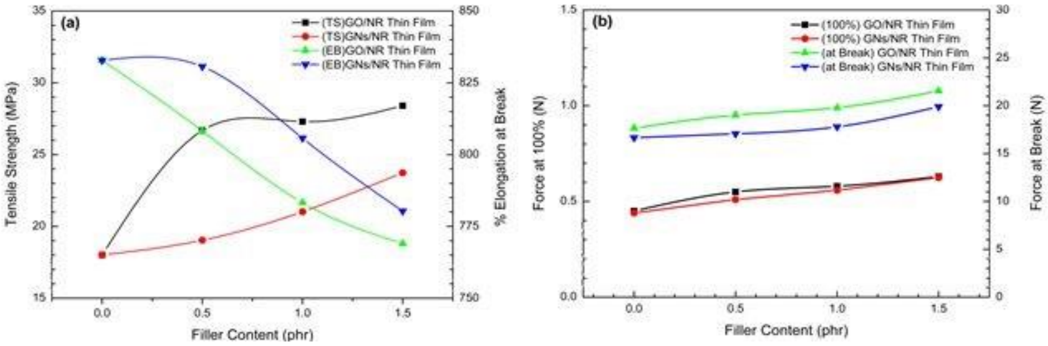


Figure 1.2 3 The (a) tensile properties (b) force at 100% and at break of filled−NR thin films[24]

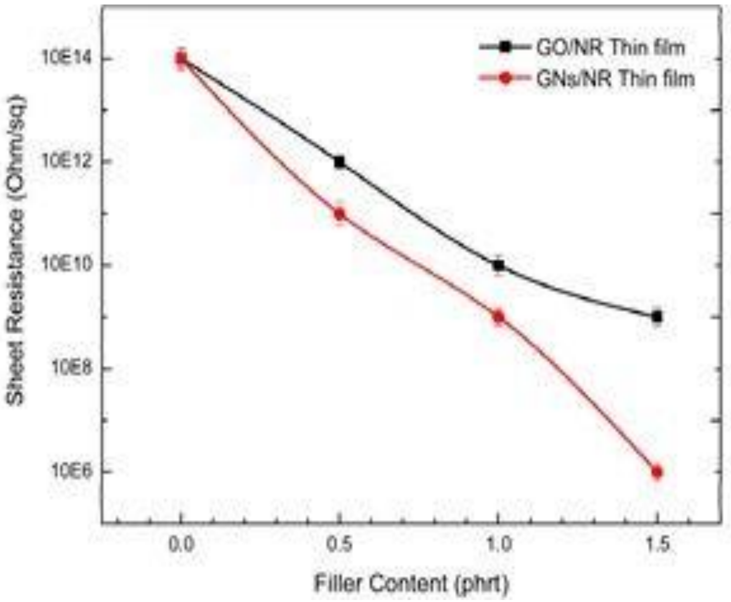


Figure 1.2 4 The sheet resistance of filled−NR composite thin films[24]

The optimum tensile strength was obtained from the 1.5 phr GO loading sample which showed 58% increment in TS compared to control NRL sample. And there were no notable changes in breaking force. This significant increase in TS compared to graphene reinforced NRL film [12] is due to the functional groups (hydroxyl, epoxy or carboxyl) which make GO strongly hydrophilic, improving the adhesion between GO and NRL and as well as their influence on the NRL vulcanization kinetic and increasing crosslink density. The sheet resistance has decreased with the increasing GO filler loading. At 1.5 phr GO loading the highest conductivity (Resistivity < 10^9 ohm/sq) was shown. We can expect this to increase with the increasing of GO loading due to incorporation of GO fillers into NRL which have superior electric properties and high aspect ratio.

1.3 Reduced Graphene Oxide as a Filler:

Chemical, photo chemical, thermal, photo thermal, microwave or microbial/bacterial methods can be utilized[4] on GO to hinder the oxygen content and to produce reduced graphene oxide(figure 1). Since the complete reduction of GO leads to perfect graphene as a product it is important to control the process to obtain rGO, although practically there will be some remaining oxygen-containing functional groups due to low possibility of all the sp3 bonds returning back to a sp2 configuration[25] . By altering the reducing methods, rGO with different carbon to oxygen ratio and different chemical compositions can be obtained.

Maria Iliut in 2016[28] and Lai Peng Lim in 2019[29] reported the incorporation of rGo into NRL. Iliut has observed the use of 0.1% rGO loading in the natural rubber latex matrix. 1 MPa increment in tensile stress at 700% elongation was stated, consequently the ultimate tensile strength of the nanocomposite was reduced with addition of rGO.

Lai Peng Lim,introduced a new method of producing rGO-NRL nanocomposite. GO was reduced to rGO without using strong reducing agents i.e hydrazine hydrate, which could affect the colloidal stability of the rGO latex mixture.

The rGO NRL dispersion was prepared using simple mixing method and coagulant dipping was performed to obtain the nanocomposite films. Produced rGO-NRL nanocomposite showed a notable improvement on physical properties and heat stability with low level loading of rGO.

The sample with just 0.1 phr loading of rGO has successfully enhanced the stress elongation at 700% (M700) and retention up to 14% and 13%, correspondingly.

It is interesting to observe that the thermal conductivity has also increased to 0.236 W/m·K or a 36% of increment with addition of very low level of rGO loading which is a very good enhancement compared to others that used a greater amount of rGO loading (> 2% or 6 phr)[12].

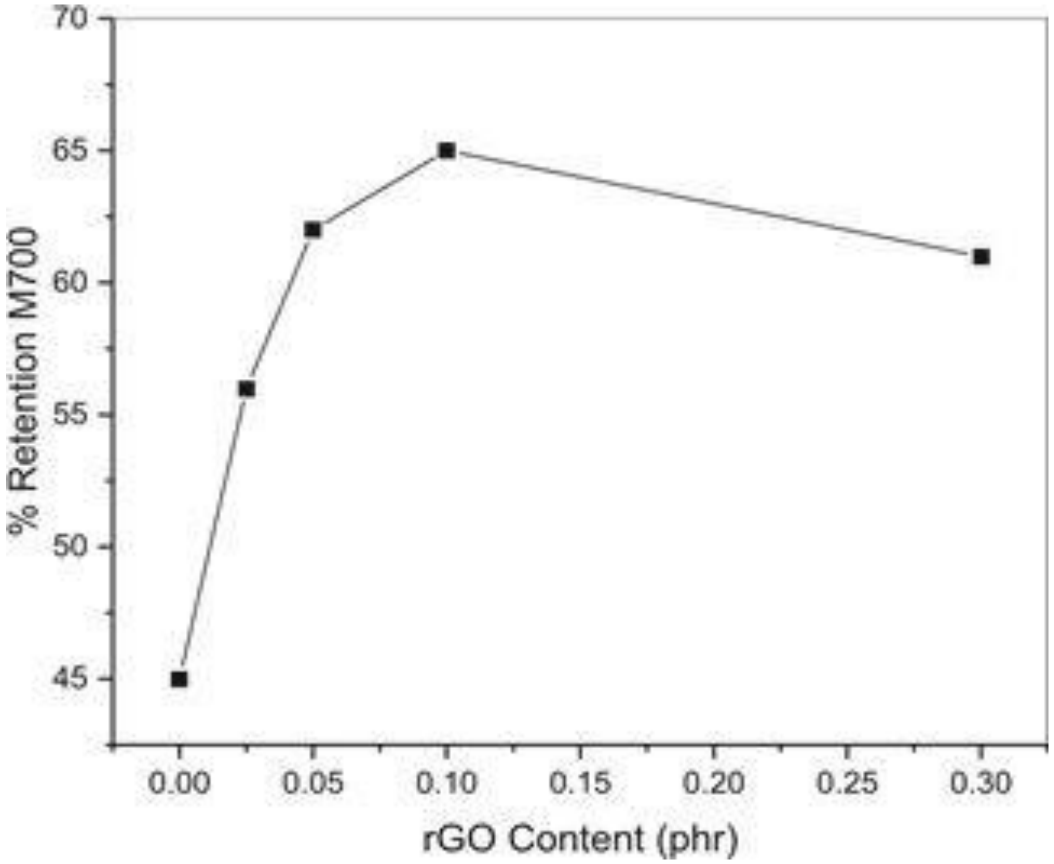


Figure 1.3 1 Percentage retention of M700 of rGO-NRL with various levels of rGO loadings[29]

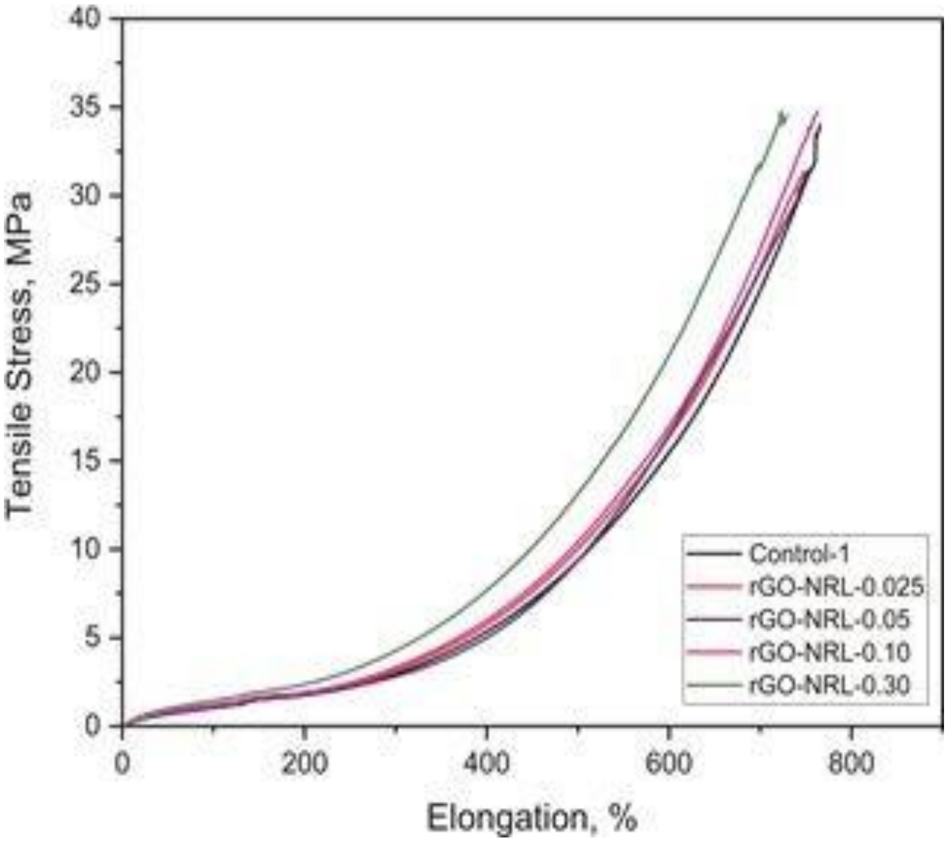


Figure 1.3 2 Stress-strain curve of rGO-NRL with various levels of rGO loadings.[29]

The resulting rGO-NRL nanocomposite showed a good enhancement on physical properties and heat stability with very slight level of rGO loading.

This improvement is attributed to the rubber-filler interactions of the rGO and natural rubber particles. Besides that, homogeneity of the composite dispersion was achieved without the use of high shear mixing or ultrasonication.

Furthermore, this method of preparation has not affected the stability of the produced rGO-NRL nanocomposite.

1.4 Graphene Nanoplates as a Filler:

Graphene nanoplates (GNPs) usually have thickness in between 1 nm and 3 nm and have lateral dimensions stretching from roughly 100 nm to 100 um[4]. GNPs are typically produced by chemical exfoliation of graphite.

Due to GNPs nanoscale size, shape, physical and chemical properties they can be used to enhance the properties on broad range of polymeric materials

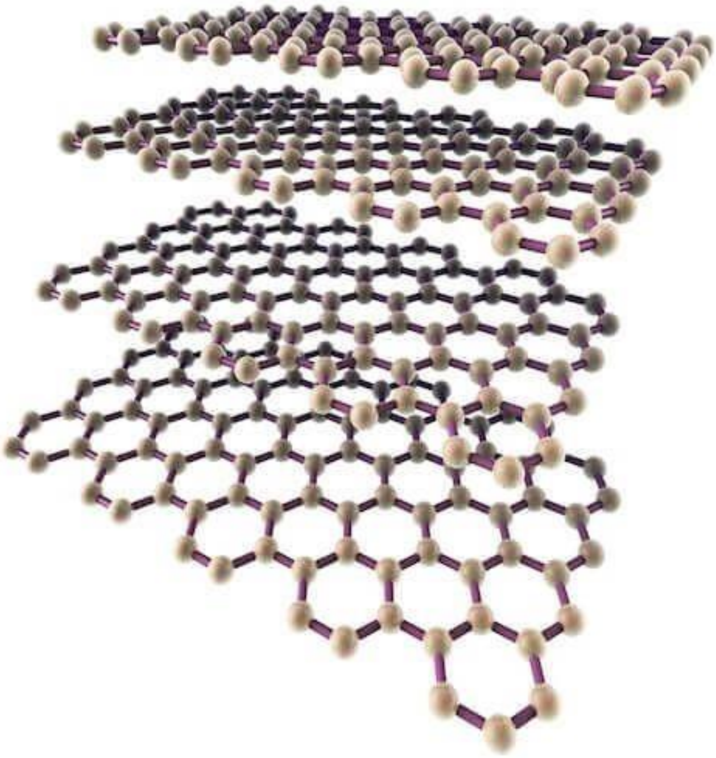


Figure 1.4 1 The GNPs structure can be envisioned as a pile of parallel layers of hexagonal carbon atoms. Due to Van der Waals interactions the sheets reagglomerate easily and have thickness of 5-20 layers

Duong Duc La [26] published an article in 2018 focusing on incorporation of graphene nanoplates (GNPs) into NRL film manufacturing. GNPs were mass-produced by a one-step chemical exfoliation of natural graphite[27] and used as a filler for the fabrication of natural rubber composite by a simple mixing method.

The GNPs obtained by one-step method were made into an aqueous solution by sonication and then simple mixing by sonication was used to produce the GNPs/NRL mixture, then it coagulated to obtain thin films.

The resultant GNPs/rubber composite was characterized by using scanning electron microscopy (SEM), and a rheometer. The synthesized graphene nanoplates had a thickness of less than 10 nm and a lateral size of tens of microns.

INSTRON 5582 tensile testing machine was used to determine the tensile and tear strength properties of the samples and GOTECH 7012 abrasion resistance tester was used to measure the wear resistance of the samples.

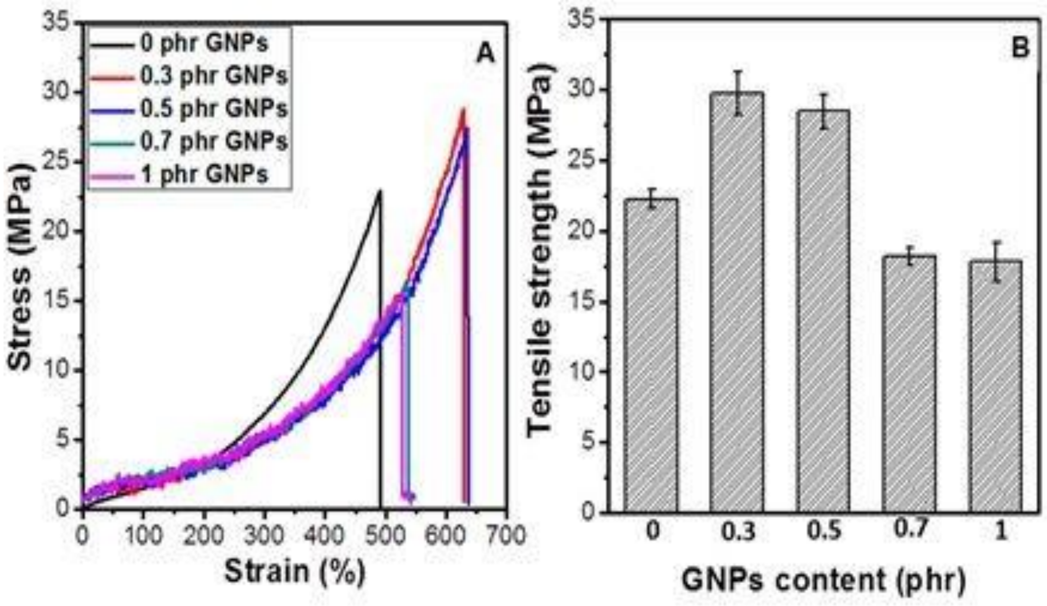


Figure 1.4 2 : (A) Stress-strain curves of all the samples and (B) tensile strength properties of GNPs/NR composites.[26]

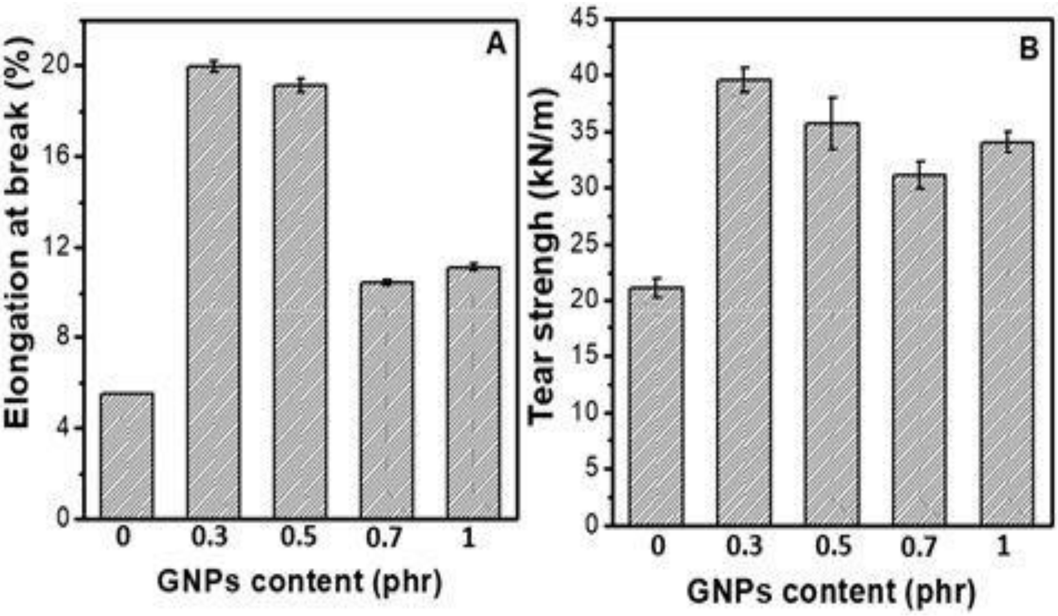
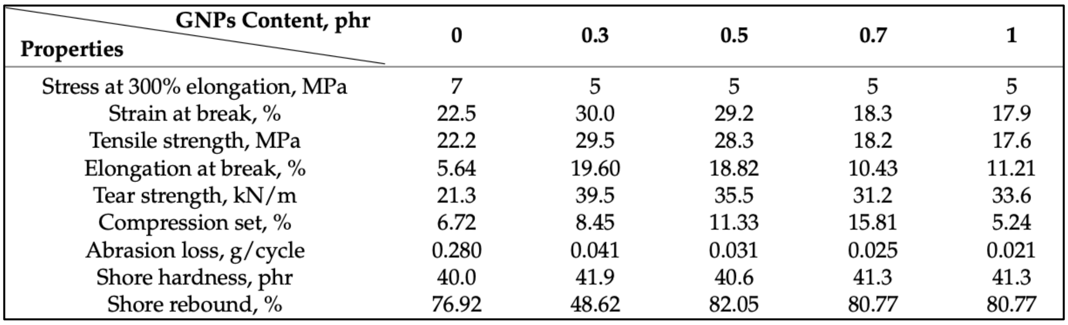


Figure 1.4 3 (A) Elongation at break and (B) tear strength of the samples[26]

Table 1 A summary of the mechanical properties of the GNPs/natural rubber composite.



GNPs Content, phr / Properties	0	0.3	0.5	0.7	1
Stress at 300% elongation, MPa	7	5	5	5	5
Strain at break, %	22.5	30.0	29.2	18.3	17.9
Tensile strength, MPa	22.2	29.5	28.3	18.2	17.6
Elongation at break, %	5.64	19.60	18.82	10.43	11.21
Tear strength, kN/m	21.3	39.5	35.5	31.2	33.6
Compression set, %	6.72	8.45	11.33	15.81	5.24
Abrasion loss, g/cycle	0.280	0.041	0.031	0.025	0.021
Shore hardness, phr	40.0	41.9	40.6	41.3	41.3
Shore rebound, %	76.92	48.62	82.05	80.77	80.77

With the GNPs reinforcing effect the tensile strength, tear strength and elongation at break were significantly increased by 30%, 200% and 400% respectively. Furthermore the rubber composite revealed an exceptional improvement of abrasion resistance loss up to seven to ten-fold. And also both the abrasion loss, effect on the hardness and the rebound properties have decreased and compression set, max torque were increased.

Conclusions:

The change of Natural Rubber Latex film properties while varying the filler from Graphene and Graphene based materials are explained in this review. Graphene and Graphene based materials are multi-functional reinforcement material which can improve the mechanical, electrical and thermal properties in NRL. This can be done in very low loadings due to the very high surface area of such materials and are very beneficial in many applications.

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