# APPLICATION OF MULTILAYER CARBON NANOTUBES (MWCNT) ON NINE TROPICAL WOOD OF COSTA RICA AND THEIR IMPACT ON MECHANICAL RESISTANCE

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#### SUMMARY

Multilayer carbon nanotubes (MWCNT) have been used in the reinforcement of materials due to their versatility; however, only little is known about their use in tropical wood. In the present study, functionalized MWCNT were introduced a hydroxyl group in water solution at two concentrations, 0.05% and 0.10%; and injected using the vacuum pressure method commonly used in wood preservation. Nine important tree species to Costa Rica i.e. Acacia mangium, Cedrela odorata, Cordia alliodora, Enterolobium cyclocarpum, Gmelina arborea, Goethalsia meiantha, Ochroma pyramidale, Tectona grandis, and Vochysia ferruginea were tested for the effectiveness of MWCNT on five types of mechanical strength. As a result of absorption studies, Vochysia ferruginea was found to absorb the most with 455.45L/m<sup>3</sup> at 0.05% and 466.40L/m<sup>3</sup> at 0.10%, while Cedrela odorata was the least absorbing species with 215.78L/m<sup>3</sup> at 0.05% and 215.78L/m<sup>3</sup> at 0.10%. However, no pattern of improvement in either of the species was detected as per expectation, which could be due to several reasons such as the origin of the material, functionalization with hydroxyl group nanotubes or the chemical composition of the species.

# Introduction

Carbon nanotubes (CNTs) are carbon allotropes such as diamond, graphite or fullerenes, and its structure can be like a graphene sheet rolled on itself (Lariza et al., 2012). They are cylinders of one or more layers with open or closed ends (De Volder et al., 2013), can have a diameter of nanometers and their length can reach up to 1mm, so the ratio of length-width is very high (Pérez, 2010). The commercial interest in CNTs is reflected in its production capacity, which exceeds several thousand tons per vear.

The characteristic advantages of CNTs like high mechanical strength and good thermal stability (Orináková and Oriňák, 2011) make them important for a perfect scientific environment product (Pérez, 2010). However, the disadvantages involve adoption of regular shapes like pentagons and heptagons during mass production, which is not the perfect union of the CNT and hence the properties can be degraded (De Volder et al., 2013). Moreover, lack of solubility in organic solvents or aqueous solutions is also a drawback of these structures (De Volder et al., 2013), but they can undergo chemical reactions that make them soluble in various solvents, and can be integrated with inorganic, organic and biological systems (Saeb et al., 2015), a process known as 'functionalization' of the nanotubes.

CNTs have several applications and can be utilized at different sectors. In medicine, they can be used for diagnosis and treatment of cancer, nervous system disorders and infectious diseases (Zhang *et al.*, 2010). They can be exploited in compound materials as electrical conductive fillers in plastics; to improve fiber compounds among wind turbine blades or vanes boats; in paints to help reducing biofouling of ship hulls (De Volder *et al.*, 2013); and very importantly as an anticorrosive coating for metals to improve the rigidity of coating (De Volder *et al.*, 2013).

On the other hand, the complex structure of the wood cell and the physical and mechanical properties of its wall has attracted the attention of scientists, and has recently led to the study of the effects of nanotechnology on wood structure (Moon *et al.*; 2006; Kamel, 2009). Among many studies, the major concern has been evaluating the possibility of working with wood in a nano scale to have more absorbent, lightweight and strong products (Shmulsky and Jones, 2011). For example. Kordkheili et al. (2012) have studied the influence of mixture of cement, wood fiber and CNT's, and they found that the static bending was higher when a mixture of 1.5% multilayer nanotubes (MWCNT), 20% fiber and 43% of cement is used. Another study, in Ficus hispida, has shown the improvement in structural properties (Hazarika and Maji, 2014). This type of wood grows in the tropics, it is soft and is not suitable for structural applications due to their poor physical and mechanical properties. However, hydroxyl group multilayered nanotubes (MWCNT-OH) in different concentrations were added to wood in a solution of ethanol

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# APLICACIÓN DE NANOTUBOS MULTICAPA EN LA RESISTENCIA MECÁNICA DE NUEVE MADERAS COMERCIALES DE COSTA RICA

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### RESUMEN

Los nanotubos de carbono multicapa (MWCNT) han sido utilizados en el reforzamiento de materiales debido a su versatilidad; sin embargo, se conoce poco sobre su uso en maderas tropicales. En el presente estudio, MWCNT funcionalizados con un grupo hidroxilo en una solución de agua a dos concentraciones (0,05 y 0,10%) e inyectado en la madera por medio de una bomba de presión comúnmente utilizada en preservación de madera. En nueve especies de importancia forestal en Costa Rica (Acacia mangium, Cedrela odorata, Cordia alliodora, Enterolobium cyclocarpum, Gmelina arborea, Goethalsia meiantha, Ochroma pyramidale, Tectona grandis y Vochysia ferruginea) se ensayó la efectividad de los MWCNT en cinco tipos de resistencia mecánica. Los resultados mostraron que Vochysia ferruginea fue la especie que más absorbió, 455,45L/m<sup>3</sup> en la concentración de 0,05% y 466,40L/m<sup>3</sup> en la concentración de 0,10%, en tanto que Cedrela odorata fue la especie que menos absorbió, 215.78L/m<sup>3</sup> en la concentración de 0,05% and 215L/m<sup>3</sup> en la concentración de 0,10%. Sin embargo, no se detectó un patrón de mejoría en cualquiera de las especies, como se esperaba. Este comportamiento puede atribuirse a varias razones, entre las que destaca origen del material, funcionalización con hidroxilo en los nanotubos o la composición química de la especie.

# APLICAÇÃO DE NANOTUBOS MULTICAMADA EM RESISTÊNCIA MECÂNICA DE NOVE MADEIRAS COMERCIAIS DA COSTA RICA

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# RESUMO

Os nanotubos de carbono de paredes multiplas (MWCNT) têm sido utilizados para o reforço de materiais devido a sua versatilidade; no entanto, se conhece pouco sobre seu uso em madeiras tropicais. No presente estudo, MWCNT funcionalizados com um grupo de hidroxila em soluções de água com duas concentrações (0,05% e 0,10%) e injetado na madeira por meio de uma bomba de pressão geralmente utilizada na preservação da madeira. Em nove espécies de importância florestal na Costa Rica (Acacia mangium, Cedrela odorata, Cordia alliodora, Enterolobium cyclocarpum, Gmelina arborea, Goethalsia meiantha, Ochroma pyramidale, Tectona grandis e Vochysia ferruginea) foi ensaiada a efetividade dos MWCNT em cinco tipos de resistência mecânica. Os resultados mostraram que Vochysia ferruginea foi a espécie com maior absorção, 455,45L/m<sup>3</sup> na concentração de 0,05% e 466,40L/m<sup>3</sup> na concentração de 0,10%, em quanto que Cedrela odorata foi a espécie com menor absorção, 215.78L/ m<sup>3</sup> na concentração de 0,05% e 215L/m<sup>3</sup> na concentração de 0,10%. No entanto, não foi detectado um padrão de melhora em qualquer uma das espécies, como era esperado. Este comportamento pode atribuir-se a varias razões, entre as quais destaca a origem do material, funcionalização com hidroxila nos nanotubos ou a composição química da espécie.

and potassium hydroxide mixed with a copolymer of melamine and formaldehyde (MFFA) in order to determine their influence on the wood structure. The hardness properties, bending and tension of the wood were found highest when the concentration of the MWCNT-OH was 1.5, which coincides with the study conducted by Fu et al. (2010). They also found that the thermal stability and flammability properties of polypropylene (PP)/wood flour enhances these properties when CNTs were added.

In Costa Rica, 17.5% of wood is used in the manufacture of furniture and an important part of the latter is exported (ONF, 2013). It is estimated that about 15 different wood species are used for manufacturing of furniture and these woods comes from plantation or natural and secondary forests stands (ONF, 2013). However, these species are of medium density and in some cases have limited resistance (Serrano and Moya, 2011), so better strength and performance with MWCNT may be an option and can add value to these woods.

The aim of this work was to study the mechanical properties (static bending, tension parallel to the fiber, lateral and axial hardness, compression perpendicular to grain, shear parallel to grain) of nine tropical species by introducing a solution of multilayer carbon nanotubes functionalized with hydroxyl groups (MWCNT-OH) at two concentrations (0.05 and 0.10%).

# **Materials and Methods**

# Materials

Multilayer carbon nanotubes functionalized with hydroxyl groups (MWCNT's-OH) were supplied by CheapTubes Inc. (Cambridgeport, MA, USA). The manufacturer's technical information for nanotubes is: outer diameter of about 50nm, length of 10 to 20 $\mu$ m, purity of 95% and an OH group concentration of 0.5-1.0% by weight.

Nine wood species used in this study were grown in tropical area of Costa Rica. They are cataloged in three groups; i) the species most used in reforestation programs: *Gmelina arborea*, *Tectona grandis* (teak) and *Acacia mangium*, usually employed in lightweight construction; ii) species used in furniture manufacturing: Cordia alliodora, Cedrela odorata and Enterolobium cyclocarpum; iii) species providing potential wood for furniture and construction and in high abundance in the secondary forest: Vochysia ferruginea, Ochroma pyramidale and Goethalsia meiantha. These woods were obtained from sawmills and timber shops near the facilities of the Technological Institute of Costa Rica.

Wood boards used presented a thickness of 6cm and in most cases the width was corresponding to the diameter of the log, which is commonly used as sawing pattern for furniture manufacturing (Serrano and Moya, 2011). The boards were air dried and 45 samples were prepared for each type of mechanical test, according to the ASTM D-143 (ASTM, 2014) standard: compression perpendicular to grain:  $2.5 \times 2.5 \times 10.0$  cm; static bending:  $2.5 \times 2.5 \times 41$  cm; shear parallel to grain:  $5.0 \times 5.0 \times$ 6.3 cm; lateral and axial hardness:  $5 \times 5 \times 15.0$  cm; and tension parallel to grain: test pieces  $2.5 \times 2.5 \times 46.0$  cm.

# Characterization of multilayer carbon nanotubes functionalized

The detail of the features. dimensions and other aspects of MWCNT-OH is widely explained by Moya et al. (2015a) and can be summarized as: the MWCNT-OH have the 3-band characteristics of multilayer nanotubes, called D, G, G' (Figure 1), and it differs from non-functionalized nanotubes in the band D. This difference is attributed to defects in the walls of the nanotubes or atoms that have been added to the structure of the nanotubes, and the differences in the bands G and G' are produced by stretching in the plane of carbon-carbon bonds in the leaves of graphene, and it also indicates disorders and defects in carbon nanotubes. Concerning the thermal stability, the MWCNT-OH used have the maximum decomposition at ~600°C, showing that they have better thermal stability than the non-functionalized nanotubes (Figure 1b).

# Preparation of the MWCNT-OH solution

MWCNT-OH solutions were prepared by adding 0.03g and

0.015g of MWCNT-OH in 200ml of water for 0.10% and 0.05% concentration, respectively. The prepared MWCNT-OH solutions were sonicated for 5min with an Ultrasonic Processor Model CV18 at an amplitude of 75% at intervals of 45sec, with10sec breaks. Then, 200ml of the solution was poured into 3L of water and re-zoned for 25min. The process continued until 15L of solution is left and finally, water was added to have 30 L of total solution.

# Impregnation of wood with MWCNT-OH

Samples were placed in an experimental pressure tank commonly used for wood preservation. The process of impregnation of MWCNT-OH solution consist of 30min at -78kPa vacuum, 2h at 690kPa pressure and 15min at -78kPa vacuum. Then, the wet samples were placed in a controlled environment for six months to reach an equilibrium moisture content of 12% (air dried wood).

# Absorption and retention-OH MWCNT in wood samples

Wood samples were weighed before and after the impregnation process and two parameters were generated: absorption capacity and retention of MWC NT-OH. The absorption capacity for each sample was calculated as the absorption of the solution in liters per timber volume (Eq. 1), while retaining MWCNT-OH absorption was determined considering MWC NT-OH by wood weight, according to Eq. 2.



# Mechanical strength tests

The effect of MWCNT-OH in improving resistance of wood was evaluated by five mechanical tests: static bending, tension parallel to the fiber, lateral and axial hardness, compression perpendicular to grain and shear parallelto grain. These mechanical tests were conducted according to ASTM D-143 standard (ASTM, 2014). For each type of mechanical test, total of 45 samples were used, 15 for each type of MWCNT-OH concentration. The bending, tension parallel to the fiber and hardness tests were performed in a universal testing machine (Tinius Olsen model H10KT) while the cutting tests and compression were carried out in a universal testing machine (Tinius Olsen model LT60).

# Statistical analysis

A descriptive analysis of the parameters studied (with each trial) was performed (mean and standard deviation). Additionally, it verifies whether the variables satisfy the assumptions of normal distribution, homogeneity of variances and the presence of extreme data. Subsequently, an analysis of variance (ANOVA) was applied to verify the effect of the concentration of MWCNT-OH on the mechanical properties studied. Then, the statistical differences between the average were established by a Tukey test at a confidence level of 95%. For that, Statistical Analysis Software (SAS) was used and a comparison was made between the means, whereas figures and tables were made in Excel 2013.

#### Results

# *MWCNT-OH absorption and retention*

C. alliodora, G. meiantha and O. pyramidale woods showed greater absorption and retention of MWCNT-OH (Table I). O. Pyramidale retained the most MWCNT-OH among the nine species in the treatment of 0.10% whereas T. grandis showed lowest uptake and retention in both MWCNT-OH concentrations.

#### Static bending

For this test, O. pyramidale, Vochysia ferruginea, E. cyclocar-

#### TABLE I RETENTION AND ABSORPTION OF NANOPARTICLES IN NINE TROPICAL SPECIES OF COSTA RICA FOR TWO CONCENTRATIONS OF MWCNT'S-OH



Figure 1. Raman spectrum (532 nm excitation) (a) and thermographic analysis for MWCNT'sy MWCNT's-OH. Fuente: Moya *et al.* (2015a)

I WO CONCENTRATIONS OF MWCNT S-OH								
Species	Absorption of MWCNT's-OH (L <sub>solution</sub> /m <sup>3</sup> <sub>wood</sub> )		Retention of MWCNT's-OH (g <sub>nanotubes</sub> /m <sup>3</sup> <sub>wood</sub> )		Wood density (kg/m <sup>3</sup> )			
	0.05%	0.10%	0.05%	0.10%	,			
Acacia magnium	361	316	1.80	3.16	533			
Cedrela odorata	218	214	1.08	2.14	391			
Cordia alliodora	435	512	2.18	5.12	346			
Enterolobium cyclocarpum	307	328	1.54	3.28	346			
Gmelina arborea	283	257	1.42	2.57	495			
Goethalsia meiantha	447	593	2.24	5.93	399			
Ochroma pyramidale	380	266	1.90	2.67	124			
Tectona grandis	271	311	1.36	3.11	596			
Vochysia ferrugia	455	466	2.28	4.66	544			

pum, C. alliodora, G. arborea and T. grandis showed no significant difference in any of the MWCNT-OH concentrations (Figure 2), while A. magnium, C. odorata and G. *meiantha* exhibited statistical differences. In A. mangium wood, the modulus of elasticity (MOE) of the wood treated with MWCNT's-OH 0.05% has lower compared to control, whereas with the 0.10% treatment no significant difference was found when compared with untreated wood. The same applies to the modulus of rupture (MOR) of wood treated with 0.05% MWCNT-OH; the MOR was statistically lower than the wood without nanotubes. In C. odorata, differences were found when nanotubes were present in the wood. Statistically, MOR of the timber with 0.05% MWCNT-OH was greater than the wood with 0.10% of nanotubes. However, there was no MOR alteration noticed with either of concentration of nanotubes, compared to untreated wood. In G. meiantha, MOR value was statistically higher in wood treated with 0.10% MWCNT-OH; however, the wood treated with 0.05% MWCNT-OH showed no difference in comparison with untreated wood.

# Tension parallel to the fiber

The ANOVA indicated that the species A. magnium, C. alliodora, E. cyclocarpum, G. arborea, O. pyramidale and V. ferruginea showed no significant differences when applied to the different concentrations, while the rest of the species differed (Table II). In C. odorata both treatments had higher values than the control. The treatment of 0.05% had a statistically higher value than the control in the case of G. meiantha, while the 0.10% treatment did not show differences with the control. Similarly, in T. grandis, the 0.05% treatment showed a significant higher value compared to the other treatment and control.

# Lateral and axial hardness

In this mechanical test, A. magnium, V. ferruginea, G. meiantha, G. arborea and C. alliodora showed significant differences in at least one of the concentrations of MWCNT-OH (Figure 3). A. magnium presented greater lateral hardness in the concentration of 0.05%, while 0.10% did not show a difference with the wood without MWCNT-OH. Whereas Vochysia ferruginea



Spacing	Stress (MPa)				
Species	0%	0.05%	0.10%		
Acacia magnium	95.87 A	87.97 A	79.55 A		
Cedrela odorata	37.30 B	69.91 A	77.02 A		
Cordia alliodora	62.13 A	66.06 A	63.19 A		
Enterolobium cyclocarpum	30.59 A	31.51 A	35.84 A		
Gmelina arborea	66.56 A	65.59 A	61.49 A		
Goethalsia meiantha	57.07 B	75.79 A	61.99 B		
Ochroma pyramidale	30.75 A	26.32 A	31.01 A		
Tectona grandis	95.63 B	151.99 A	113.59 B		
Vochysia ferruginea	45.92 A	44.69 A	40.78 A		

Average values identified by the letters A and B are statistically different at  $\alpha = 95\%.$ 

presented difference in axial hardness. Wood treated with 0.10% MWCNT-OH had less lateral stiffness than the one treated with 0.05% concentration MWCNT-OH. However, G. meiantha presented significantly higher values with the 0.10% concentration of MW CNT-OH and C. alliodora had differences in both lateral and axial hardness in both cases: the treatment of 0.05% had a statistically higher value than the untreated wood. While Gmelina arborea also differ in lateral and axial hardness, in this case the differences are in the treatment of 0.10%, which was statistically greater than wood without MWCNT-OH (Figure 3).

# Compression perpendicular to grain

In the compression test, G. arborea, G. meiantha, and O. pyramidale were the only species with significant differences between treatments (Table 3). Gmelina arborea presented a statistically higher value when wood was treated with MWCNT-OH at a concentration of 0.05%, and O. pyramidale and G. meiantha were statistically observed with lower value in treated wood (MWCNT- OH concentration of 0.05%).

# Shear parallel to grain

In this strength test, *A. magnium* and *C. alliodora* showed some significant differences (Figure 4). In both the species, when wood is treated with MWCNT-OH to a concentration of 0.05%, a significant increase in strength was observed relative to the wood with 0.10% treatment and without MWCNT-OH (Figure 4).

# Discussion

# Absorption and retention

The behavior of increase in absorption (Table I) with the decrease in wood density is consistent with results of other studies where some form of nanoparticles were used in wood (Moya *et al.*, 2014, 2017). Consistent with the above, it was also found that a greater amount of MWCNT/m<sup>3</sup> was absorbed in lower density



Figure 2. Average effort (MPa) modules of rupture (MOR) and modulus of elasticity (MOE) for nine tropical wood with two concentrations MWCNT's-OH (0.05% and 0.10%). Average values identified by the letters A and B are statistically different at  $\alpha = 95\%$ .



Figure 3. Mean axial and lateral hardness in nine tropical wood species with different concentrations MWCNT's-OH. Average values identified by the letters A and B are statistically different at  $\alpha = 99\%$ .

TABLE III EFFORT IN COMPRESSION PARALLEL TO GRAIN IN NINE TROPICAL WOOD SPECIES WITH DIFFERENT CONCENTRATIONS OF MWCNT'S-OH

Creasian	Stress (MPa)				
Species	0%	0.05%	0.10%		
Acacia magnium	44.27 A	45.95 A	42.57 A		
Cedrela odorata	37.68 A	38.31 A	38.99 A		
Cordia alliodora	36.06 A	31.85 A	35.24 A		
Enterolobium cyclocarpum	29.41 A	33.58 A	33.70 A		
Gmelina arborea	37.21 B	48.75 A	40.69 B		
Goethalsia meiantha	35.89 AB	30.73 B	36.84 A		
Ochroma pyramidale	11.48 A	6.60 B	7.23 B		
Tectona grandis	40.83 A	42.11 A	41.55 A		
Vochysia ferrugia	28.89 A	25.81 A	27.59 A		

Note: Average values identified by the letters A and B are statistically different at  $\alpha$ = 99%.

wood, such as *O. pyramidale*, *C. alliodora* and *G. meiantha*, while species like *T. grandis* and *A. mangium*, with higher density, the absorption was lower and retained less of MWCNT (Table I).

*V. ferruginea* had the highest absorption value, unlike *O. pyramidale*, of low density, which did not present the same absorption as the previous species. The low retention of MWCNT in *T. grandis* or *A. mangium* wood is explained by the fact that this wood has a high percentage of heartwood, which is not possible to preserve. Moya *et al.* (2017) reported that in the vacuumpressure treatment, penetration is null when there is heartwood; but there is adequate sapwood penetration in these wooden species.

# Mechanical tests

Although several authors had shown that the use of MWCNT-OH improves wood properties (Hazarika and Maji, 2014; Kordkheili *et al.*, 2012), the results of this study were not consistent with them, as most species included show no significant improvement in the mechanical tests performed. This behavior can be attributed to the MWCNT-OH having imperfections or impurities, which do not allow a suitable combination with wood substrates (De Volder, 2013), so this situation cannot allow adequate support of MWCNT-OH and wood.

Another aspect that may be contributing to the ineffectiveness of the strengthening of tropical wood with the injection of MWCNT-OH is the type of functionalization used. The hydroxyl group used in this work to functionalize MWCNT is commonly used as a precursor (Xie *et al.*, 2010) that increases the strength of wood by adding another functional group compatible to wood components.

Although the carboxyl functional group allows interaction with the four hydroxyl groups of the cell wall polymers in the wood fiber, this is used as a crosslinking agent to modify the wood fibers (Xie et al., 2010). The results in the different woods resistance were not as effective. Also, influencing the ineffectiveness of the MWCNT-OH is the variability and complexity of the chemical composition of these tropical species, which also varies between and within species (Kilic and Niemz, 2012; Moya *et al.*, 2015b), so that the functional group of nanotubes might not have a proper interaction with the timber.

Finally, the irregularity presented in the mechanical testing in two concentrations of MWCNT-OH is also an important parameter to demonstrate. For example, G. arborea wood exhibited a greater resistance at 0.05% concentration in comparison to 0.10% in some experiments, but the hardness of the same species showed the opposite effect, the stress value in the 0.05% treatment being higher than 0.10%. This irregularity can be attributed to variability in the wood used. Those samples were extracted from different timber shops, and these are usually not separated according to the different parts of the tree. The fastgrowing plantations of wood in tropical regions generally have greater variation in wood properties (Moya and Muñoz, 2010).

#### Conclusions

The vacuum-pressure method can be used for MWCNT injection into wood; however, the hydroxyl group functionalization of the nanotubes is not suitable for a reinforcement of the mechanical properties of the nine commercial species of Costa Rica studied. Furthermore, the strength values of these nine species were not homogenous and it was not possible to establish an effect of MWCNT. With the objective of knowing the effects of MWCNT on wood, it is necessary to standardize some aspects, such as wood precedence. It is also most important to use a type of functionalization that allows greater interaction of MWCNT with wood structure.

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Figure 4. Shear stress parallel to the grain of nine tropical wood species with different concentrations MWCNT's-OH. Average values identified by the letters A and B are statistically different at  $\alpha$ = 95%.

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