

USE OF CARBON NANOTUBES TO ENHANCE
THE PHYSICAL AND MECHANICAL
PROPERTIES OF WOOD COMPOSITES

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MECHANICAL PROPERTIES OF WOOD COMPOSITES

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ABSTRACT

Medium Density Fiberboard had been widely used in the furniture construction. Malaysia is one of the develop country which is produce MDF board but less competitive due to lack of mechanical properties. This research is about to increase the productivity of Medium Density Fiberboard (MDF) by mixing the Carbon Nanotubes (CNT) as filler. The Carbon Nanotubes would increase the thermal and mechanical properties of MDF. The main purpose of this research is to enhance the mechanical properties of the wood composite boards and the surface smoothness of the boards. Some of the problems for using Carbon Nanotubes are the uniform dispersion in Urea Formaldehyde. To solve that problem, the Carbon Nanotubes have to undergo treatment with acid solution. The treated CNTs can be used in the manufacturing of MDF boards. The different amount of CNTs will be used for comparing the results. The result shows that, the increasing amount of CNTs had improved the MOR, IB and Thickness Swelling values. The 5.0% of CNTs show the greatest value of MOR, IB and Thickness Swelling. So it proved that the CNTs can improve the mechanical properties of wood composites. As a conclusion, the improvement of mechanical properties of wood composites will affect the production of wood composites in Malaysia. Moreover, it should increase the demand of wood composites and the uses of wood composites around Malaysia and can be exported outside Malaysia.

ABSTRAK

Papan Sederhana Tumpat telah banyak di gunakan dalam penghasilan perabot. Malaysia telah menjadi salah satu negara yang mengeluarkan Papan Sederhana Tumpat tetapi kurang mendapat saingan disebabkan kekurangan daya tahan. Projek ini bertujuan untuk meningkatkan produktiviti Papan Sederhana Tumpat dengan mencampurkan serbuk karbon nano sebagai penguat. Serbuk karbon nano dapat meningkatkan pengendalian haba dan kekuatan papan ini. Tujuan utama projek ini adalah untuk meningkatkan kekuatan komposit kayu dan juga kelicinan papan kayu ini. Walaubagaimanapun, terdapat satu masalah berkaitan serbuk karbon nano iaitu serakan seragam di dalam Urea Formaldehyde. Untuk selesaikan masalah ini, serbuk karbon nano akan menjalani rawatan menggunakan larutan asidik. Serbuk karbon yang di rawat boleh digunakan dalam proses pembuatan Papan Sederhana Tumpat. Jumlah serbuk karbon yang berbeza akan digunakan di dalam setiap sampel Papan Sederhana Tumpat untuk membandingkan setiap bacaan yang terhasil. Hasil daripada ujian kekuatan menunjukkan peningkatan pada MOR, IB dan pengembangan ketebalan dengan meningkat bilangan serbuk karbon nano. Papan yang mengandungi 5.0% serbuk karbon nano, menunjukkan kadar peningkatan yang tinggi bagi ujian MOR, IB dan pengembangan ketebalan. Kesimpulannya, peningkatan sifat kekuatan komposit kayu akan memberi kesan kepada penghasilannya di Malaysia. Tambahan lagi, ia akan meningkatkan lagi permintaan komposit kayu dan penggunaannya di serata Malaysia dan juga boleh dieksport ke luar Malaysia.

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LIST OF SYMBOLS

W_{wet}	Initial weight
W_{dry}	Final weight
P	Peak Load
L	Length
b	Width
a	Thickness
w	Width
l	Length
T_f	Final thickness
T_i	Initial thickness
M_1	Molarity
V_1	Volume
V_{HNO3}	Volume of acid nitric
V_{water}	Volume of water
ρ	Density
m	Mass
MOR_{avg}	Average of Modulus of Rapture
IB_{avg}	Average of Internal Bonding
ω	Weight
$TS\%_{avg}$	Average of Thickness Swelling
V_{UF}	Volume of Urea Formaldehyde

LIST OF ABBREVIATIONS

MPIC	Ministry of Plantation Industries and Commodities Malaysia
MTIB	Malaysia Timber Industries Board
FIDEC	Fiber and Bio composite Development Centre
CNTs	Carbon naotubes
MWCNTs	Multiwalled Carbon Nanotubes
SWCNTs	Single Wall Carbon Nanotubes
MDF	Medium Density Fiberboard
UF	Urea Formaldehyde
MOR	Modulus of Rupture
IB	Internal Bonding
TS	Thickness swelling
UTM	Universal Testing Machine
G	Gmelina arborea
L	Leucaena leucocephala
2 h	Two hours
15 h	Fifteen hours
MC	Moisture Content

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Wood composites are a panel prepared by pressing a fiber mixed with thermosetting resin. Usually, fiber board is derived from wood by certain process. It will be used hot pressing to get a panel with a desired product. Generally panel has specific thickness between 3 mm to 40 mm. Moreover, this thing had been used widely and had found extensive markets in the furniture and cabinetry industries. Malaysia is one of leading country doing exports furniture around world.

Bio Composite Industry is one of top leading industries in Malaysia. It was established by The Ministry of Plantation Industries and Commodities Malaysia (MPIC) and Malaysia Timber Industries Board (MTIB). It was the Fiber and Bio composite Development Centre (FIDEC). Bio composites is prepared by mixing the natural fiber as a filler to enhance the mechanical properties and others properties to support the bio composites. Their objectives are to facilitate pre-commercialization of fiber and bio composite material, to stimulate interest in and to enhance the utilization of fiber and bio-composite materials in the country and to coordinate the overall development of the fiber and bio-composite industry in Malaysia. MTIB has been major source of earnings to contribute the nation's economic growth. In this new era, challenges for wood based industries comes from the competitiveness of productivity, production cost and advance product development.

However, this research will be conducted by using nano particle to produce new type of composites. The nano particles were used in order to minimize the usage of natural fiber as filler. Nano-wood composites are one of the new technologies developments on improving wood products. Actually, the nano-wood composites are a panel prepared by mixing wood fibers, nano particles with thermosetting resin. It will work as a replacement for wood based composites. In future, this technologies will be accepted in Malaysia to have high competitive with other develop country.

In this case, Carbon Naotubes will be added in the wood composites. Carbon Nanotubes have outstanding mechanical, electrical, and thermal properties. Carbon Nanotubes (CNTs) are one of the most famous things that had been attract more scientists to do research on it. Usually, the Multiwalled Carbon Nanotubes (MWCNTs) is widely use in the research compared to Single Wall Carbon Nanotubes (SWCNTs). It is affected by the cost for synthesizing the SWCNTs is expensive compared to MWCNTs.

In Malaysia, there were 14 plants available to produce Medium Density Fiberboard (MDF) board. In 2010, they had export MDF around 12.0 million and it makes Malaysia as top three of world largest exporting MDF after Germany and France. Medium Density Fiberboard (MDF) also can be classified as hardboard and it is 100% of wood fiber glued under pressure and temperature for curing. Medium Density Fiberboard is usually used for furniture construction because it gives many benefits that led it to be popular. Other than that, this furniture construction is supplied to school, home and office furniture. The accessories include the construction of desks, high quality marker boards, work surfaces, pillars, and other products. MDF board had been trusted to be used because of very strong, provides longevity, and is resistant to warping that occurs over time, effect by moisture. This type of board was chosen as the samples to be tested in these nano-composites technologies.

Based on internet sources, it state that the consumer benefit with cost savings compared to real wood, because MDF boards are man-made from wood fibers, rather than particles (About MDF Board - Medium Density Fiberboard is often used as backing for Marker Boards, 2011). In addition, there are some other advantages for

MDF which are give the smoothness on the surface and that makes it easy to apply paint, varnish and laminates directly to the surface. Moreover, compared to plywood and chipboard, MDF had been chosen based on the physical properties, which is dense, flat, stiff, has no knot and easily to machine. The reason why it is easily being cut caused of made by fine particle and prevents it from forming grain on its surface. The grain would give damages on the cutting surface. Other than that, MDF board also can be drilled, machined and filed without damaging the surface if there is no grain formed. Every advantage will be embroidered with disadvantages. MDF contains Urea Formaldehyde (UF) which can be released during sanding and cutting process. This action should be taken proper safety to conduct the MDF or otherwise it could be harmless and dangerous.

Developments on the base MDF are needed to compete with other MDF production companies. The newest technology to improve the properties of MDF is by adding some additives. In other terms are making the wood composites. Composites can be defined as a material made by two or more constituent with significantly different in chemical and physical properties which remain separated. In term of wood composites, there are two term usually been used which are matrix phase and fiber. Based on those two terms, the wood fibers as matrix phase and fiber used is Carbon Nanotubes. Matrix usually used to bind the fibers together so that the applied stress is distributed to all fibers. Other than that, it is used to protect the surface of the fibers from damage and to separate the fibers and restrain from crack propagation. The crack propagation will minimize the properties of the composites. Other than that, the fiber must be strong, have high mechanical properties to support the stress applied to the composite.

Moreover, the strengthening mechanism involves in the interaction of the matrix and the fiber, which is between the wood fibers and the CNTs. The matrix must be ductile and absorb the load from outside. If the fiber and matrix have good interaction, so the transmittance of the load can be distribute nicely and produce high mechanical strength. The dispersing agent also needed in order to create good interaction between wood fibers, Urea Formaldehyde and the CNTs.

1.2 IDENTIFICATION PROBLEM

This research had been conducted due to lack of mechanical properties in the wood production. Mechanical properties can be minimized if the curing is not complete due to the distribution of heat through the wood fibers. It is caused by the Urea Formaldehyde did not cured well. Urea Formaldehyde is thermosetting resin which is can be brittle after curing. In the hot pressing, the thermosetting resin will be cured and make the fiberboard become dense and brittle. The curing time of urea Formaldehyde will affect on the fibers to bind together and minimize the wood fibers pull out that will minimize the mechanical properties. Second is, cost of production is high in the mean time boards are less competitive in the international market. Someday, the Bio composites can be replaced by nano-composites to be more competitive in market. Finally is, low mechanical properties such as bending strength and internal bonding. This properties will affect the qualities of wood composites (MDF) produced. The most major problem is the dispersion of the CNTs in the organic solvents or in organic solvents. It have the stable bond so it not easily to form another bond without any dispersing agent or treatment. If this problem encountered, so it is easy to produce the wood composites with CNTs. Then, the mechanical properties should be inherited by the MDF board.

1.3 RATIONALE AND SIGNIFICANCE

Based on the properties of carbon nanotubes, it should give advantages to the wood composites (MDF) after mixing it in the wood composites in the process. Firstly, higher conductive carbon nanotubes will increase the heating and cooling rate of the composite. "Some literature said that, thermal conductivity of an isolated SWCNT to be as high 6600W/mK at room temperature" (Han Gi Chae, Jing Liu, and Satish Kumar, 2006). Other than that, higher chance to spreading uniformly among woods fiber, caused by it sizes. Moreover, carbon nanotubes and grapite will enhance the surface smoothness along with other mechanical properties. It is also will not damage the circular saw, while cutting and other furniture making process. If the matrix and fibers disperse well, it will inherit the mechanical strength of the CNT. This effect could

produce high productivity of the MDF board and increase the demand of MDF for making the furniture.

1.4 RESEARCH OBJECTIVES

Objective for this research are:

RO1: To enhance the mechanical properties of wood composites.

RO2: To improve the surface smoothness of the boards.

RO3: To increase the production of MDF board in a year.

1.5 SCOPES OF RESEARCH

This part will discuss more specific part in this process, which means the method will be run to produce it. The major equipment is hot pressing, which is used to produce the lab scale of MDF board. Hot Pressing will be used to compress the wood fiber to desired thickness and density. Before that process, the CNTs will be treating in the acid solution based on literature. The treatment used to modify the CNTs, so that it will disperse in the adhesive (Urea Formaldehyde). After that, the adhesive will be sprayed to the wood composites before going into hot pressing. At the end, all the MDF samples will be tested for Modulus of Rupture (MOR), Internal Bonding (IB) and Thickness swelling (TS). All testes will be done by using Universal Testing Machine (UTM) by using standard method for testing wood. Then, the result will be compared with the standard board in order to satisfy the objectives of this research.

CHAPTER 2

LITERATURE REVIEW

2.1 BIO-COMPOSITE INDUSTRY

In order to further develop the fiber and bio-composite industries in Malaysia, the Ministry of Plantation Industries and Commodities Malaysia (MPIC) and Malaysian Timber Industry Board (MTIB) established the Fiber and Bio-composite Development Centre (FIDEC) in Selangor, Malaysia. This company also do Research and Development on searching the newest technique to improve the properties of bio composites (Developing the fibre and biocomposite). In other time, the nano-composite will be exposed in Malaysia same like bio-composites. The natural fiber will be substitute with the nano particle in order to enhance the properties of wood panel.

2.2 COMPOSITES

Composite is combination of two or more different material. “Most of composites have been created to improve combinations of mechanical characteristics such as stiffness, toughness and ambient and high temperature strength” (Callister, W. D. & Rethwisch, D. G., 2008). “Many composite materials are composed of just two phases. One is termed the matrix, which is continuous and surrounded the other phase, often called the dispersed phase” (Callister, W. D. & Rethwisch, D. G., 2008). In this research, the wood fiber can be characterized it as the matrix because it is used to surround the Carbon nanotubes (CNTs). The matrix used to protect the surface of the fiber from any defect caused by external forces. It would cause of minimizing the strength of the fiber that would lead to crack propagation.

2.3 WOOD COMPOSITE

This article was reviewed about the effect of MOR and IB on the cement-bonded wood composite board. In this article was testing on two types of exotic tropical hardwood species, *Gmelina arborea* (G) and *Leucaena leucocephala* (L) and a mix of the two species at equal percentage (G + L). The parameter used to test was different density can effect the MOR and IB. Results from this reseach was shown in Table 2.1 below.

Table 2.1: Modulus of Rupture and Internal Bonding of cement-bonded flakeboards Influenced by board density.

Species	Density (kg/m ³)	MOR (N/mm ²)	IB (N/mm ²)
Gmelia Arborea	1000	7.68	0.16
	1100	8.84	0.12
	1200	9.75	0.11
Leucuena leucocephala	1000	5.00	0.08
	1100	5.15	0.07
	1200	8.67	0.08
Gmelia + Leucuena	1000	5.49	0.12
	1100	8.12	0.10
	1200	8.93	0.08

Source: Ajayi, B., 2005

“Lower compression ratio and level of inter-flakes contact in L board resulting in weaker bonds and the presence of voids probably accounted for the lower strenght of the boards.”(Ajayi, B., 2005). Good compression on the manufacturing is important in order to make good contact between the wood fibers. “The low IB strength of L board maybe attributed to coarse, rough surfaces and the presence of voids within the core of boards. In the case of G board, flakes were larger and well compressed within boards, which aid better inter-flakes surface contact and little or no voids within boards. Board surfaces were also smoother and all of these factors may have contributed to the higher strength of the G board compared with L and G + L boards” (Ajayi, B., 2005).

2.4 CARBON NANO TUBES

Carbon nano tubes (CNTs) are one of the most famous things that had been attract more scientists to do research on it. CNTs have a smaller size but can give a magnificent benefit to many scientists. “CNTs is relate to it size of diameter, which is approximately 50,000 times smaller than the width of a human air but it can be up to several micrometer in length.” (Seetharamappa, J., Yellappa, S. and D’Souza, F., 2006). Moreover, “CNTs were categorized as sp^2 bond because of built up by sp^2 hybridized carbon atoms and now attracting scientists from various disciplines due to their fascinating physico-chemical properties” (Endo, M., Hayashi, T., Kim, Y. A., Terrones, M. and Dresselhaus, M. S., 2004). The scientist also respect about it properties even it size is smaller. “The cylindrical molecules with novel properties which are can produce outstanding mechanical, electrical, thermal and chemical properties. Some evidence noted in article state that CNTs 100 times stronger than steel and thermal conductivity comparable to that diamond.” (Seetharamappa, J., Yellappa, S. and D’Souza, F., 2006). If CNTs reinforced-wood composites, the physical and mechanical properties will be increase. That’s why CNTs have attracted the fancy of many scientists worldwide. In addition, “CNTs can be devided into two type, which are Single-walled nanotube (SWCNT) and Multi-walled Nanotube (MWCNT). SWCNTs was discovered in 1993, most of SWCNTs have a diameter close to 1 nm, with a tube length that may be many thousands of times larger and up to orders of centimeters.” (Zhu, H. W., Xu, C. L., Wu, D. H., and Wei, B. Q., 2002). Some said that, SWCNT can be conceptualized by wrapping single atom into a cylinder, mean that it has only one thick layer of graphite make a shape like a cylinder (Figure 2.1 a). Meanwhile, MWCNTs described as a multiple layer of graphite in on themselves to form a tube shape with interlayer spacing of 3.4 Å (Figure 2.1 b). Another specification is about the size of MWCNT which is describing in a range from 1 to 50 nm of outer diameter while the inner is usually of several nanometers.

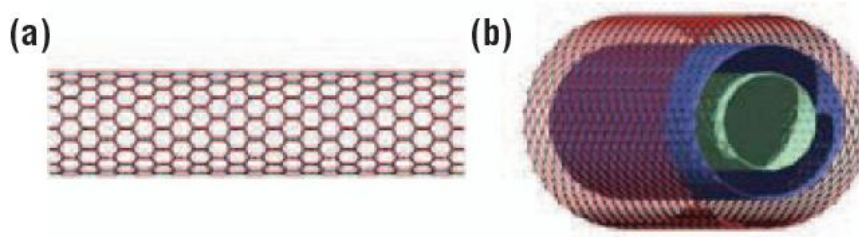


Figure 2.1: a) Single-Walled Carbon Nanotube (SWCNT)
b) Multiple-Walled Carbon Nanotubes (MWCNT)

Source: Jaldappagari Seetharamappa, Shivaraj Yellappa, and Francis D'Souza, 2006

Before this had said that “CNTs had an outstanding mechanical strength it is due to a strong carbon–carbon covalent bond are highly dependent upon the atomic structure of nanotubes and the number of shells” (Endo, M., Hayashi, T., Kim, Y. A., Terrones, M. and Dresselhaus, M. S., 2004). Below is the table of comparison the mechanical strength of CNTs with other material.

Table 2.2: The comparison of mechanical strength of CNTs with other material.

Material	Young Modulus (GPa)	Tensile Strength (GPa)	Density (g/cm ³)
Single wall nonotube	1054,000	150,000	
Multiwall nanotube	1200,000	150,000	2.600
Steel	208,000	0.400	7.800
Epoxy	3,500	0.005	1.250
Wood	16,000	0.008	0.600

Source: Seetharamappa, J., Yellappa, S. and D'Souza, F., 2006

Therefore, “SWCNTs and MWCNTs have been studied intensively as fillers in various matrices, especially polymers.” (Ajayan, P. M., Stephan, O., Colliex, C. & Trauth, D, 1994). The purpose is to make a better composite reinforce with nanotubes. The composites will be affected by CNTs properties. “Just like this research, use CNTs to enhance the mechanical and physical properties of wood composites. The thermal conductivity of an isolated SWCNT to be as high 6600W/mK at room temperature.” (Chae, H. G., Liu, J. and Kumar, S., 2006).

Table 2.3: Comparison table of Thermal Conductivity

Material	Thermal Conductivity (W/m –K)
Copper	390
Silver	430
Diamond	2600
Carbon Nanotubes	6600

Source: Konesky, 2006

“Moreover, a single-walled carbon nanotube (SWNT), which lack of imperfection, serves as the ultimate reinforcing fiber with regard to its strength in the direction of the nanotube axis” (Lafdi, 2010). “Unfortunately, SWNTs are difficult and expensive to manufacture. The difficulties in synthesizing SWNTs have caused researches to pursue multi-walled carbon nanotubes (MWNTs) and carbon nanofibers as a viable substitute for the SWNTs for many applications.” (Saito, 1998).

Other properties of CNTs had been state that it was built by carbon-carbon structural arrangement and affected the mechanical, electrical and thermal properties. (Lau, 2010) “Because of that, carbon nano co-structured materials like carbon blacks and nanotubes have been well accepted as the strongest nano-reinforcement for composite material.” (Lau, 2010)

2.5 RESIN AND WOOD TREATMENT

Resin is one of the main parts in order to gluing the wood fiber together. From that it will give a desired shape inside the hot pressing. Moreover, in the manufacture of the wood compositions, curing is usually effected by applying a heat treatment. Examples of curable resins that are widely used in the manufacture of wood composites include urea formaldehyde resins and phenol formaldehyde resins. For my research I will be used urea formaldehyde resins to be as adhesive in the wood composite (Martinus L. Van Druten, 1997).

Wood is extensively used in modern society in particular for housing materials and furniture. “However, the woods tend to contain defects such as warp, spiral grain, crooks, breaks, strains, rot and also engaged in using wood, such defects constitute an extremely serious problem.” (Motai, 1978). Usually, it used heat treatment for curing the wood.

2.6 UREA FORMALDEHYDE

“Urea-formaldehyde resins are formed by the reaction of urea and formaldehyde.” (Conner, 1996). “The overall reaction of urea with formaldehyde is quite complex and, although initially studied early in this century, is not completely understood at the present time.” (Conner, 1996) In this research, the Urea Formaldehyde is used in the production of MDF as glue. The bond between the wood fibers and and resin will be cured by usinh the hot press. This Urea Formaldehyde known to be widely used in the industrial of making board. It cans be prove in the literature, “which is more than 70% of this urea-formaldehyde resin is used by the forest products industry for a variety of purposes.” (Conner, 1996). “The resin is used in the production of an adhesive for bonding particleboard (61%) of the urea-formaldehyde used by the industry), medium density fiberboard (27%), hardwood plywood (5%), and a laminating adhesive for bonding (7%), for example, furniture case goods, overlays to panels, and interior flush doors.” (Conner, 1996). The Urea Formaldehyde also known as the thermosetting resin which is consists of cross-linkage bond. Thermosetting is the irreversible plastic which is cannot re-melted and after the curing it become brittle. There are outnumber of

advantages, which are including low cost, ease of use under a wide variety of curing conditions, low cure temperatures, water solubility, resistance to microorganisms and to abrasion, hardness, excellent thermal properties, and lack of color, especially of the cured resin (Conner, 1996).

2.7 PURIFICATION OF CARBON NANOTUBES

The Carbon Nanotubes (CNTs) known well that possess good mechanical, electrical and thermal properties. It is due to the carbon-carbon bond generated in the CNTs. The disadvantage of using CNTs is insoluble in the liquid. It can be proved in many literatures that used CNTs as part of their research. “Disaggregation and uniform dispersion are critical challenges that must be met to successfully produce such high property materials, since carbon nanotubes tend to self-associate into micro-scale aggregates.” (Vaisman, L., Wagner, H. D. & Marom, G., 2006)

Purification methods can be done in various ways. It can be divide into three methods, which are chemical, physical, and a combination of both. “Basically, the chemical method purifies CNTs based on the idea of selective oxidation, wherein carbonaceous impurities are oxidized at a faster rate than CNTs, and the dissolution of metallic impurities by acids.” (Vaisman, L., Wagner, H. D. & Marom, G., 2006). “The physical method separates CNTs from impurities based on the differences in their physical size, aspect ratio, gravity, and magnetic properties, etc. In general, the physical method is used to remove graphitic sheets, carbon nanospheres (CNSs), aggregates or separate CNTs with different diameter/length ratios. In principle, this method does not require oxidation, and therefore prevents CNTs from severe damage.” (Vaisman, L., Wagner, H. D. & Marom, G., 2006). This treatment will lead to the removal of the impurities on the carbon surface. Later then it will be accepted to disperse in the solvents.

The purification using chemical can be divided into three other ways, which are in gas phase, liquid phase and electrochemical. Each one consists of different methods of purification. Moreover, for the physical also can be divided into six others solution method which are using filtration, centrifugation, solubilization with functional group,

high temperature annealing, chromatography and others method to remove metal particles. Final method is combination which is also having different method under it. It can be divided into four methods, which are:

- (i) HIDE, wet grinding, filtration, oxidation, sonication, centrifugation
- (ii) Filtration/magnetic filtration, oxidation, annealing
- (iii) Sonication in H_2O_2 , $\text{HNO}_3/\text{HF}/\text{SDS}$, filtration
- (iv) High temperature annealing extraction.

All these method will give the modified surface of CNTs in order to disperse in the solvent.

The liquid chemical treatment method was using acid treatment in order to make the functionalize group of-COOH on the CNTs surface. This functionalization was used by other researcher in order to improve carbon nanotubes (CNTs) interaction and dispersion. "MWCNTs is more widely carried out by treatment with aqueous solutions of acids or oxidizing agents, such as H_2SO_4 , HNO_3 , $\text{H}_2\text{SO}_4/\text{HNO}_3$, H_2O_2 , KMnO_4 , $\text{KMnO}_4/\text{H}_2\text{SO}_4$, and $(\text{NH}_4)_2\text{S}_2\text{O}_8$." (Chiang, Y. C., Lin, W. H. & Chang Y. C., 2011). In the same articles state that, if the amount of acidic group on the carbon surface it will reverse the effect of the hydrophobicity of the CNTs. In other words, it will decrease the properties of hydrophobicity, hence making the metal precursors in aqueous solution more accessible to the surface and resulting in more metal deposition. There are many journal talk about the treatment of CNTs using acid treatment. From this treatment it is change the bond of CNTs from SP^2 to SP^3 . This method can be done for a short period time because the acid used can be found in laboratory which is HNO_3 and H_2SO_4 . It can be done for Nitric acid treatment only or can be in mixture of $\text{H}_2\text{SO}_4/\text{HNO}_3$ (3:1).

The method using was almost the same. "In the first procedure (CNTsnc), CNTs were immersed in a mixture of $\text{H}_2\text{SO}_4/\text{HNO}_3$ (3:1) at room temperature. CNTs were treated in an ultrasound bath for 2 h and upheld for 15 h. The chloridric acid was, then, added to the solution. Subsequently, this solution was neutralized with ammonium hydroxide and filtered with a 0.22 mm cellulose acetate membrane. The CNTs were