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## Analyzing the dispersion of carbon nanotubes solution for use in Portland cement concrete

C. G. N. Marcondes<sup>1</sup>; M. H. F. Medeiros<sup>2</sup>

- <sup>1</sup> Pontifical Catholic University of Paraná (PUCPR), Brazil.
- <sup>2</sup> Department of Civil Engineering, Federal University of Paraná, Brazil.

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alconpat.int@gmail.com, Website: www.alconpat.org

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## **ABSTRACT**

Carbon nanotubes (CNTs) are nanometric carbon structures with cylindrical formats. For use in concretes, one of the difficulties is in its dispersion, focus this work. It used a tool known as hierarchical analysis to investigate the efficiency of the dispersion of carbon nanotubes in concrete kneading water. Were studied 12 forms of dispersions in aqueous medium containing hum Miscellaneous Chemicals. Carbon nanotubes multi-walled in powder form and Processed already dispersed in water were used. The study showed that the hierarchical analysis tool might constitute an alternative to the election of the best choice among the available options, considering the factors of influence in a systemic way.

Keywords: analytical hierarchy process; concrete; carbon nanotubes

#### **RESUMO**

Os nanotubos de carbono (CNT) são estruturas nanometricas de carbono com formatos cilíndricos. Para uso em concretos, uma das dificuldades está na sua dispersão, foco deste trabalho. Foi usada uma ferramenta conhecida como análise hierárquica. Para investigar a eficiência da dispersão dos nanotubos de carbono na água de amassamento do concreto, foram estudados 12 formas de dispersões em um meio aquoso contendo diversos produtos químicos. Foram utilizados os nanotubos de carbono de paredes múltiplas em forma de pó e os industrializados, já dispersos em água. O trabalho demonstrou que a ferramenta de análise hierárquica poderia se constituir em uma alternativa eficiente para a eleição da melhor dispersão, considerando os fatores de influência de forma sistêmica.

Palavras-chave: análise hierárquica; noncreto; nanotubos de carbono.

## RESUMEN

Los nanotubos de carbono (CNT) son estructuras nanométricas de carbono en formas cilíndricas. Para su uso en hormigón, una de las dificultades es su dispersión, enfoque de este trabajo. Se utilizó una herramienta conocida como análisis jerárquico para investigar la eficiencia de dispersión de los NTC en el agua de la mezcla de hormigón. Fueron estudiados 12 maneras de dispersiones en medio acuoso que contiene diferentes productos químicos. Se fue utilizado los nanotubos de carbono de pared múltiple en forma de polvo y los ya procesados, dispersos en agua. El estudio mostró que la herramienta de análisis jerárquico podría constituir una alternativa eficaz para la elección de una mejor dispersión, teniendo en cuenta los factores que influyen en forma sistémica.

Palabras clave: proceso de análisis jerárquico; hormigón; nanotubos de carbón.

Autor de contacto: Marcelo Medeiros (medeiros.ufpr@gmail.com)

## 1. INTRODUCTION

Research in cement and nanotechnology areas, such as Makar et al. (2005), Gleize (2007), Nochaiya e Chaopanich (2011), have shown that some nano-composite additions on cement may allow important changes in the cementitious composites properties, allowing the production of stronger cements, less porous and durable. Among these possible nano materials are carbon nanotubes (CNT), the main focus of this paper.

Carbon nanotubes (CNTs) are carbon structures that, once synthesized, get cylindrical shape in nanometer scale and measure approximately 3nm in diameter and 1000nm in length, one nanometer corresponds to 10E-9 meter (Couto, 2006).

From the structural point of view, there are two types of carbon nanotubes: single wall, consisted of a single graphene sheet rolled up on itself to form a cylindrical tube, and multiple walls, comprising a set of coaxial carbon nanotubes, with several graphene sheets rolled up into a tube (Zarbin, 2007). Single-walled carbon nanotubes (SWNTs) are more difficult to be synthesized, which increases their cost and virtually precludes its application in large scale.

The addition of CNTs to cementitious compounds is a topic that has been studied by various brazilian and international universities and the justification for this is that some surveys, attest the good performance of nanoparticles when added to Portland cement mortars and pastes. However, there are still some barriers to overcome. According to Batiston (2012), the two main challenges for the introduction of carbon nanotubes in cementitious matrices are: homogenizing the distribution of carbon nanotubes in the matrix and tailoring the interaction of CNTs with the resulting compounds from the hydration of cement.

Regarding the distribution of CNTs to a cementitious matrix, several methods have been used, highlighting the sonification and functionalization of CNTs using nitric and sulfuric acid mixture. An appropriate dispersion of carbon nanotubes (CNTs) is a prerequisite for their use in improving the mechanical properties of cement-based composites (Sobolkina et al., 2012).

For researchers Koshio et al. (2001), ultrasound can be considered an effective technology for dispersing carbon nanotubes in water, oil or polymers. For them, the shear forces generated by ultrasound outweigh the attraction forces between the nanotube particles, being able to separate them. This has been proven by Konsta et al. (2010) who achieved an efficient dispersion by application of ultrasonic energy and the use of a surfactant. The results of this research show that there was adequate dispersion with the application of ultrasonic energy. It was also evident in this study that multi-walled carbon nanotubes can reinforce cement matrix, while increasing the amount of CSH and reducing porosity. This contribution is also due to the fineness of the particles, which results in a reduction of the pore size of the hydrated cement paste (Neville, 1996).

As the use of carbon nanotubes in cement compounds is a field of study in its early stages and promising development, this study aims to contribute to a better understanding regarding the carbon nanotube dispersion efficiency in aqueous solutions. In order to choose the best additions to perform the dispersion was employed hierarchical analysis tool, which will be presented below, and has the advantage of being a systematic method of choice, in which several criteria can be evaluated in a comprehensive manner.

## 2. THEORY OF HIERARCHICAL ANALYSIS

The Analytical Hierarchic Process, AHP, is one of the multi-criteria analysis methodologies that assists the decision making in several fields of human knowledge. In civil engineering, the potential of AHP has been little explored, however it is possible to mention examples of studies that have used this tool as a decision making tool, such as: Marchezetti et al. (2011), in the treatment of household waste; Lai et al. (2008), in public works projects; Costa and Correa (2010), in post-occupation evaluation of buildings;

Pereira, Medeiros and Levy (2012) and Mattana et al. (2012) in studies on recycling of construction waste for the manufacture of concrete and mortar.

According to Costa (2002) the method proposed by Saaty in the early 70s (Saaty, 1978), can be classified as one of the most known and used methods of multi-criteria analysis, aiming at the selection/choice of alternatives in a process that considers different evaluation criteria.

For the use of this important tool that deals with complex problems in a more simplified way, key elements are needed to determine the global target: feasible alternatives and the set of criteria and attributes. It is important that the user of the technique is aware that the established criteria do not cause a superposition effect on each other and are able to cover all solutions of the problem.

The AHP allows to consider the subjectivity of some parameters and uses forms of judgment to quantify these items, so that hierarchical levels are built. The results are presented in the format of priorities, enabling the determination of how much one alternative is superior to the other, or their degree of importance about the other variables.

This review influences the quality and effectiveness of the obtained results, since it is the evaluator's responsibility to determine the modelling of alternatives and criteria, and the judgment on the assigned values in the involved evaluations. Requiring the evaluators knowledge about the subject of each topic.

## 3. EXPERIMENTAL PROCEDURE

The focus of the developed experiment is basically to classify a composition and a dispersion method of carbon nanotubes in aqueous solution. The reason is the fact that being the carbon nanotubes previously dispersed in water, its distribution in the concrete mass to be made will be more homogeneous than if the CNT powder is simply added to the mixer at the time of concrete manufacture. With this focus, 12 dispersion alternatives were evaluated by the point of view of turbidity, diameter of group formation and sedimentation tendency. Table 1 shows the general plan of the experiment, with the number of samples and the used dosage.

Table 1. Overview of the experiment.

Sample	Description	Dosage
SM 1	CNT in powder with water	10g water + 0.03g of CNT
SM 2	CNT in powder with water and additive based on	10g water + 0.03g of CNT +
	polycarboxylate (Tec Flow 8000 – at 2% relative	0.2g of additive
	to the mass of water)	
SM 3	CNT in powder with water and additive based on	10g water + 0.03g of CNT +
	polycarboxylate (Tec Flow 8000 – at 1% relative	0.1g of additive
	to the mass of water).	
SM 4	CNT Aquacyl 0301 with water.	9.03g water + 1g of CNT
		Aquacyl 0301
SM 5	CNT Aquacyl 0301 with water and additive based	9.03g water + 1g of CNT
	on polycarboxylate (Tec Flow 8000 - at 1%	Aquacyl 0301 + 0.1g of
	relative to the mass of water).	additive.
SM 6	CNT Aquacyl 0301 with water, CNT in powder	9.57g water + $0.5g$ of CNT
	and additive based on polycarboxylate (Tec Flow	Aquacyl 0301 + 0.015g CNT
	8000 - at 1% relative to the mass of water).	in powder + 0.1g of additive
SM 7	CNT Aquacyl 0301 with water and additive based	9.03g water + $1g$ of CNT
	on polycarboxylate (Tec Flow 8000 – at 0.5%	Aquacyl 0301 + 0.05g of
	relative to the mass of water).	additive.

SM 8	CNT Aquacyl 0301 with water, CNT in powder	10g water + 0.03g of CNT +
	and additive based on polycarboxylate (Tec Flow	0.05g of additive.
	8000 – at 0.5% relative to the mass of water).	
SM 9	CNT Aquacyl 0301 with water, and CNT in	9.57g water + 0.5g of CNT
	powder.	Aquacyl 0301 + 0.015g CNT
		in powder
SM 10	CNT Aquacyl 0301 with water, CNT in powder	9.57g water + 0.5g of CNT
	and additive based on polycarboxylate (Tec Flow	Aquacyl 0301 + 0.015g CNT
	8000 – at 0.5% relative to of water).	in powder $+ 0.05g$ off additive
SM 11	CNT in powder and gum arabic and water	9.0g water + 1g Goma arabic
		+ 0.03g CNT in powder
SM 12	CNT in powder and water with surfactant -	-10g water $+0.03g$ of CNT in
	Sodium Lauryl Ether sulfate	powder + 0.1g of surfactant.

#### 3.1. Materials

The materials employed were: carbon nanotubes (CNT), superplasticizer admixture, distilled water, Arabic gum and Lauryl ether sodium sulfate.

In the case of Arabic gum, the manufacturer does not provide data of chemical and mechanical characterization, therefore, it is not presented in this paper. The sodium Lauryl Ether sulphate is basically a chemical product which formula is CH<sub>3</sub>(CH<sub>2</sub>)10CH<sub>2</sub>(OCH<sub>2</sub>CH<sub>2</sub>)nOSO<sub>3</sub>Na. Both materials had already been searched by Ibarra et al. (2006) and Metaxa et al. (2012) respectively, which obtained good dispersion results.

The CNTs used in this research were acquired in Nanocyl SA company, located in Belgium. The amount paid for the powder product was 120 Euros per kilo. These are multi-walled CNTs synthesized by the method of chemical vapour deposition, also called CVD. Commercially the products are specified with the names NC 7000 for powder form and AQUACYL 0301, to the one already dispersed in water. Both cases were employed in this study.

Tables 2 and 3 show the chemical and physical information of CNTs used in this study.

To perform the experiment, a superplasticizer based on polycarboxylate admixture was used. Polycarboxylate are macromolecules used as dispersants in cementitious compositions of high efficiency by reducing the viscosity of the suspensions and minimizing the amount of water used for the process (Mehta; Monteiro, 2013).

Table 2. CNT characterization provided by the manufacturer.

Properties	Unity	Value
Average diameter	nanometers	9.5
Average length	micron	1.5
Carbon purity	%	90
Metal oxide	%	10
Surface area	$m^2/g$	250-300
Average density	g/l	60

Table 3. Product composition provided by the manufacturer.

Components	% (weight)
Synthesized graphite (CNT)	90%
Cobalt oxide	< 1%
Others	9%

The admixture used in the experiment consists of a carboxylic ether polymer modified with a solid concentration of approximately 49%. The product meets the requirements of ASTM C 494/2013 (TYPE A and F) (2013), ASTM 1017/2007 (2007), NBR 11768/2011 (2011). Some of its properties can be viewed in Table 4.

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Table T. Illioillandii abot	at the superplusherzi	aumature provided t	y the manufacturer.

Recommended dosage (on weight of cement)	pH (ABNT 10908)	Specific mass (ABNT 10908)		
0.3% a 2%	5.5 <u>+</u> 1,0	$1.10 \pm 0.02 \text{ g/cm}^3$		

## 3.2. Procedure for the preparation of CNT dispersion

Figure 1-a shows the weighing of nanotubes using an electronic balance accurate to 0.001g. In all cases, the percentage of 0.3% in relation to the total water added was maintained. The mixtures were made in test tubes, which were shaken in a mechanical shaker (Figure 1-b, before, and Figure 1-c, after shaking). As a next step, the solutions in the test tubes were subjected to sonication in an ultrasound bench device, from Thornton Ltd. with a nominal frequency of 40 KHz and 100W of power (Figure 1-d).



Figure 1. a) Weighing of CNTs in balance, b) appearance of the solution prior to mechanical agitation, c) appearance of sample after mechanical agitation, d) samples in ultrasound.

The sample remained in the ultrasound for 1 hour. This time was determined based on a visual analysis that indicated that after 60 minutes, the samples showed no changes regarding tonality and turbidity and showed no settling after a rest time of 24 hours, as explained in section 3.3

## 3.3. Defining the sonication time of dispersions

This part of the study was intended to determine the permanence time of the aqueous solutions, with CNT, in ultrasound. Thus, dispersions were made by using the times of 10, 20, 40 and 60 minutes in ultrasound. The objective was to set a time of sonication that would generate a minimal incidence of dispersion decantation after 24 hours of settling. Figure 2 is a comparison example of decantation after 24 hours and for 40 minutes of sonication.

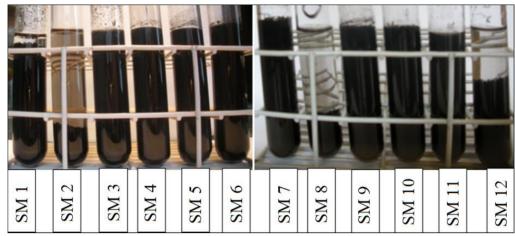


Figure 2. Visual analysis of samples with sonication time of 40 minutes after 24 hours of rest.

The result of this comparison is shown in Table 5 and indicates that sonication times of 40 and 60 minutes were those with lower occurrence of decantation. Thus, it was decided that this study would have as standard procedure to fix a time of 60 minutes for submission to sonication.

Table 5. Samples decantation after 24 hours at rest in relation to the time of ultrasonic waves' application.

Samples / Sonication time	SAM1	SM2	<b>EMS</b>	SM4	SMS	9MS	<b>ZMS</b>	8MS	6MS	SM10	SM11	SM12	Total occurrences of decantation
10 min.	Y	Y	Y	N	N	N	N	Y	N	N	Y	Y	6
20 min.	Y	Y	Y	N	N	N	N	Y	N	N	Y	Y	6
40 min.	Y	Y	N	N	N	N	N	Y	N	N	N	Y	4
60 min.	Y	Y	N	N	N	N	N	Y	N	N	N	Y	4

 $Y - \overline{Decantation\ occurred}$ 

N – Decantation did not occurred

## 3.4. Use of microscopy for dispersions evaluation

After preparing the samples of dispersions, they were analyzed in an optical reflection microscope brand Olympus, model BX60 equipped with a digital camera Olympus UC 30 that can be observed in Figure 3. Images were made with different magnifications (50x, 100x and 200x) with lighting in clear weather and lower incidence.

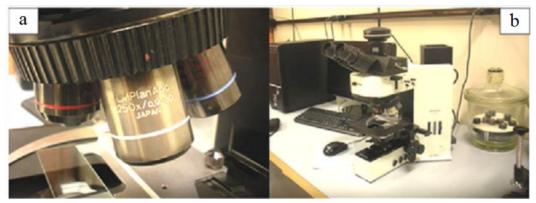


Figure 3. a) Detail of the equipment's lens b) Olympus BX 60 microscope.

Samples were collected from the test tube, immediately after preparation, before the decantation of the CNTs occurred and arranged in glass plates with the help of a pipette, dripping one drop on each plate. In all tests, it was used the incidence of illumination from down in order to examine the transparency and turbidity of the sample.

## 3.5 Evaluation Criteria

Three criteria for evaluation of dispersion of carbon nanotubes were used in this study. Turbidity, the diameter of clumps and decantation. The explanation of each of these criteria is presented below:

## 3.5.1 Turbidity

Turbidity is a physical property of fluids which results in reduction of their transparency due to the presence of suspended materials that interfere with the passage of light trough them. However, the complexity of optical interactions between incident light, optical properties of the suspended and dissolved materials, in particular its refractive index and color, turn turbidity a subjective visual property, not behaving as a directly measurable physical quantity. However, analysis of this criterion was performed in qualitatively way with the visualization or not of turbidity and its classification was made by three parameters that can be seen in Table 6. Thus, the more turbid the solution is, the more efficient the CNTs dispersion was. To determine the turbidity, it was used the incidence of light under the sample to make sure the samples were always photographed focusing the edge of the dispersed solution drop.

Incidence of light passing through the sample	Parameters	Classification
No incidence of light	Blurred	Great dispersion
Low incidence of light	Translucent	Good dispersion
High incidence of light	Translucent	Bad dispersion

Table 6. Parameters for analysis and classification of turbidity.

## 3.5.2 Diameters of clumps

The formation of clumps denotes the agglutination of particles and therefore that there was no efficient dispersion of CNT in the sample. The larger the diameter of the formed clump is, the less efficient the dispersion was. The images obtained by microscopy allowed to measure the diameter of clumps formed in each sample. To measure the clumps images increased 50 x in microscope were used, and it was agreed to measure, in microns, the diameter of the greater clump found. The measurement was made by comparison with the reference scale of the image. Figure 4 illustrates the adopted procedure.

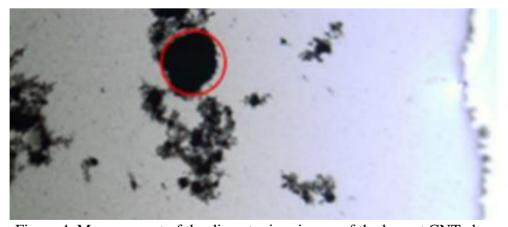


Figure 4. Measurement of the diameter in microns of the largest CNT clump.

#### 3.5.3 Decantation in test tube

Decantation is the process of separating the phases of a mixture or solution (see Figure 3). If there is considerable portion of particles in the sample, it means that there was no decantation. This is important so that there is proper dispersion of CNTs in aqueous media, and the higher the concentration of suspended particles is, the better, as for making concrete with CNTs it will be initially necessary to perform the CNTs dispersion in a liquid for later implementation.

## 4. RESULTS AND DISCUSSION

## 4.1. Evaluation of microscopy photos

In Figure 5 to 16 it is possible to view photos taken on the microscope that were used for analysis of turbidity and size of formed clumps. They have magnifications of 50x, 100x and 200x, which can be perceived in images A, B and C - respectively, in each figure. As a standardized form of images, all photos were taken using as reference one of the edges of the drop placed on a glass plate. In the photos, it is also possible to see the size of clumps formation as well as the transparency of samples with light with low incidence.

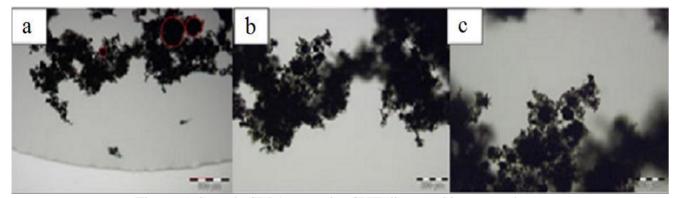


Figure 5. Sample SM 1 - powder CNT dispersed in water alone.

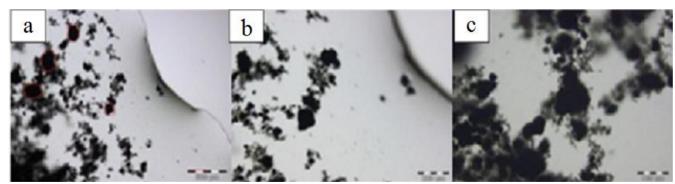


Figure 6. Sample SM 2 - powder CNT dispersed in water and a polycarboxylate admixture (Tec Flow 8000 - at 2%).

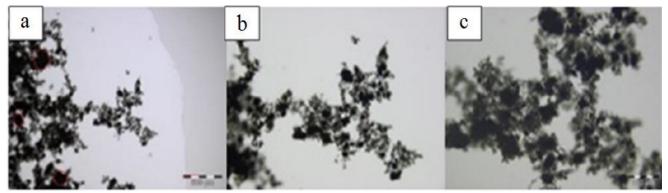


Figure 7. Sample SM 3 - powder CNT dispersed in water and a polycarboxylate admixture (Tec Flow 8000 - at 1%).

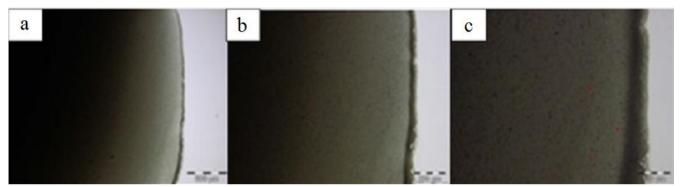


Figure 8. Sample SM 4 - CNT Aquacyl 0301 dispersed in water.

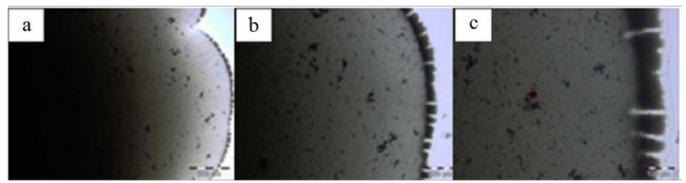


Figure 9. Sample SM 5 – CNT Aquacyl 0301 dispersed in water and polycarboxylate admixture (Tec Flow 8000 - at 1%).

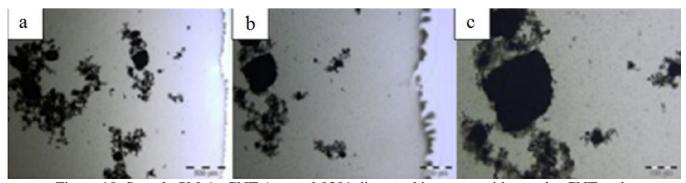


Figure 10. Sample SM 6 - CNT Aquacyl 0301 dispersed in water, with powder CNT and polycarboxylate admixture (Tec Flow 8000 - at 1%).

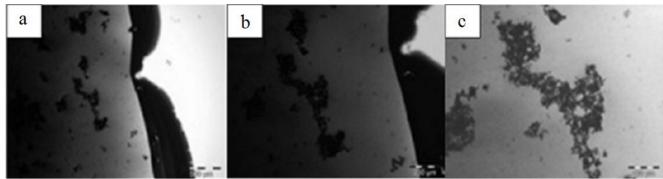


Figure 11. Sample SM 7 - CNT Aquacyl 0301 dispersed in polycarboxylate admixture (Tec Flow 8000 - at 0.5%) and water.

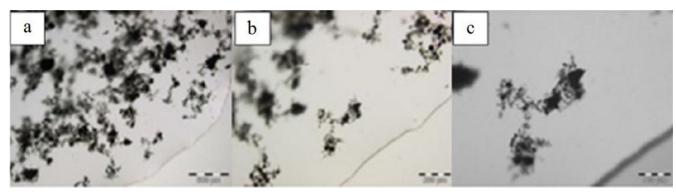


Figure 12. Sample SM 8 - powder CNT dispersed in polycarboxylate admixture (Tec Flow 8000 - at 0.5%) and water.



Figure 13. Sample SM 9 - CNT Aquacyl 0301 dispersed in water and powder CNT.

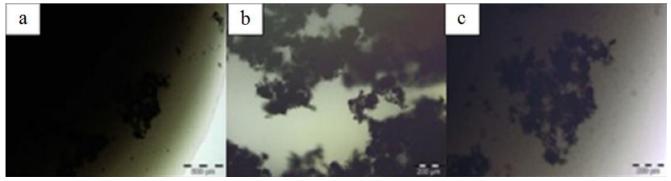


Figure 14. Sample SM 10 - CNT Aquacyl 0301 dispersed in water, with powder CNT and polycarboxylate admixture (Tec Flow 8000 - at 0.5%).

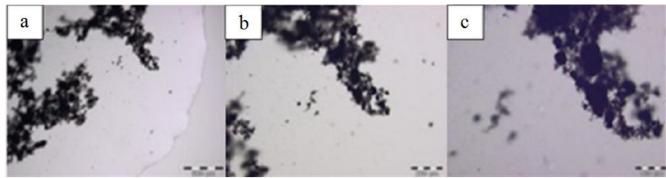


Figure 15. Sample SM 11 - powder CNT dispersed with Arabic gum and water.

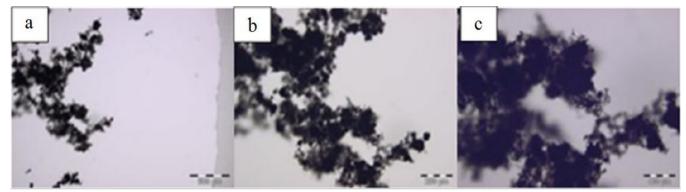


Figure 16. Sample SM 12 - powder CNT dispersed in water and surfactant.

Comparing sample SM 4 (Figure 8), which is the case of the industrially dispersed product (Aquacyl) mixed with water and subjected to 60 minutes of sonication, with the cases where were used the CNTs in powder for dispersion, it is verified that none of alternatives using powder CNT (Figures 5 to 7, Figure 10 and Figures 12 to 16) resulted in the same efficiency of the industrial dispersion of the supplier. Exceptionally, the manufacturer does not provide information about the technique used to disperse the CNTs. It is noteworthy that this difference was verified by microscopic evaluation, although many of the dispersions made with powder CNT were visually identical to SM 4, as can be seen in Figure 2. It can be seen that, in sample SM 5 (Figure 9), with superplasticizer additive TF8000 and Aquacyl product, CNTs formed some clumps and had an orientation at the edge of the drop solution. It is possible to imagine that the additive action was the cause for this occurrence, since in the sample containing only the Aquacyl and water (SM 4) this was not observed (Figure 8).

## 4.2. Application of hierarchical analysis for interpreting results

For the purposes of analyzing the efficiency of dispersion three criteria were adopted. In Figure 17 there is a general flowchart of the application of hierarchical analysis, showing that the criteria considered in the evaluation were: turbidity of dispersion, diameter of formed clumps and decantation tendency.

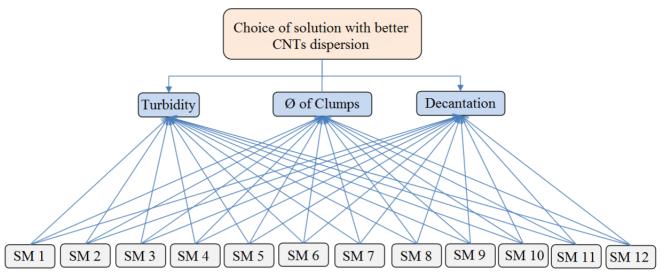


Figure 17. Flowchart of hierarchical analysis.

From the choice of this criteria, limits of performance were suggested that will be analyzed based on visual analysis and microscopy of CNT dispersions. The performance limits adopted can be seen in Table 7.

Table 7. Suggested limits for analysis of alternatives performances.

Criterion	<b>Performance limits</b>	Test method
Liquid turbidity If blurred - optimum		Microscope
-	If translucent* – good	
	If transparent** - bad	
Ø of clumps	From 0 to 500 µm – optimum	Microscope
	From 500 to 1000 µm – good	_
	Higher than 1000 µm - bad	
Decantation	Did not decanted—optimum	Visual - Test Tubes
	Decanted - bad	

<sup>\*</sup> Lets few light pass by

To estimate the importance of each criterion a matrix was developed, which can be seen in Table 8 and is contained in ASTM E 1765/2011 (2011). In the matrix, the attributes were compared by paired analysis (two by two), which aims to rank the criteria. A feature of AHP is the subjectivity of the process, since it depends of the importance that the evaluator gives to each criterion. Nevertheless, this aspect can be seen as a positive factor because it indicates that the evaluation system is open to the convictions of the decision maker, i.e., one can introduce prior experience from the expertise of the decision maker.

Regarding the use of the ASTM E 1765/2011 (2011) scale of importance, it should be clarified that when the comparison results in a reverse way to the cases of Table 8, the reverse of the note is adopted. That is, if B is more important than A, its note is 1/5.

<sup>\*\*</sup> Lets much light pass by

Table 8. Scales of importance for the evaluated criteria (ASTM E 1765/2002).

Comparisons	Scale
A equal to B	1
<b>A</b> slightly more important than <b>B</b>	3
A more important than B	5
A much more important than <b>B</b>	7
A extremely more important than B	9

The next step was to mount a decision matrix and proceed to calculate the relative weight (Pr) of each considered criterion. For this, the sum of each individual criterion is considered, dividing it by the total sum of the criteria and multiplying by one hundred. Equation 1 illustrates this calculation.

$$Pr = \frac{\sum Criterion(totalo\ frow)}{\sum Total(criteriacolumn)} \cdot 100$$
(1)

Table 9. Matrix with paired analysis and weight for each criterion.

Evaluated criteria using the importance scale according to	Turbidity	Ø of	Decanting	Criteria – Sum	Weight
ASTM E 1765/2002	of liquid	clumps	Decanting	(Line Total)	(Pr) - %
Turbidity of liquid	1.00	3.00	3.00	7.00	53.9
Ø of clumps	0.33	1.00	0.33	1.66	12.8
Decanting	0.33	3.00	1.00	4.33	33.3
Total - criteria column				13.00	100%

From the measurement of weights for each criterion, the samples were classified according to their performance. For this purpose, three levels of performance classification where agreed as shown in Table 10.

Table 10. Standard for analysis of samples.

Performance limits	Points
Comply with high performance	2
Comply averagely	1
Comply underperforming	0

Thus, after sorting the samples according to their performance, each variable was divided by its greatest value as shown in Table 11. This practice has the function of normalizing all quantities measured so that all range from 0 to 1.

Table 11. Results of samples.

Scale of		General data											Normalized data											
importance according with the analysis of CNTs dispersion	SM 1	SM 2	SM3	SM 4	S MS	9 WS	SM 7	SM 8	6 MS	SM 10	SM 11	SM 12	SM 1	SM 2	SM 3	SM 4	SM 5	SM 6	SM 7	SM 8	8M 9	SM 10	SM 11	SM 12
Turbidity of liquid	1	0	0	2	2	2	2	0	2	2	0	0	0,5	0	0	1	1	1	1	0	1	1	0	0
Ø of formed clumps (µm)	1600	1350	1100	90	180	1180	350	850	420	620	700	009	0	0	0	1	0,3	0	0.1	0.1	0.1	0.1	0.1	0.1
Decanting	0	0	2	2	2	2	2	0	2	2	2	0	0	0	1	1	1	1	1	0	1	1	1	0

Finally, each variable was multiplied by its respective relative weight, obtaining the performance index to each alternative and criterion, as shown in Table 12. Adding the data of columns in Table 12 the general index of performance of each alternative is obtained. The performance index of the 12 samples can be seen at the penultimate row of Table 12. As a result, it appears that the best dispersions were SM 4 (1st place), SM 5 (2nd place), SM 7 (3rd place) and SM 9 (3rd place). It should be noted that SM 3 and SM 11 immersions were the ones with the best performance among those who used the addition of powder CNTs.

Table 12. Alternatives performance.

	1)	Normalized data x weight of variable													
Scale of importance according to the analysis of CNTs dispersion	Variable weight	SM 1	SM 2	SM3	SM 4	SM 5	SM 6	SM 7	SM 8	6 WS	SM 10	SM 11	SM 12		
Turbidity of liquid	0.539	0.27	0.00	0.00	0.54	0.54	0.54	0.54	0.00	0.54	0.54	0.00	0,00		
Ø of formed clumps (µm)	0.128	0.00	0.00	0.01	0.13	0.04	0.01	0.02	0.01	0.02	0.01	0.01	0,01		
Decanting	0.333	0.00	0.00	0.33	0.33	0.33	0.33	0.33	0.00	0.33	0.33	0.33	0,00		
Total	0.27	0.00	0.34	1.00	0.91	0.88	0.89	0.01	0.89	0.88	0.34	0.01			
Classification	6	7	5	1	2	4	3	8	3	4	5	8			

## 5. CONCLUSIONS

The highest value found corresponds to the best alternative of choice, so it can be said that in this case, SM 4, containing water and Aquacyl 0301 CNT, corresponds to the sample with the best dispersion. This demonstrates that the industrially dispersion method is really more efficient than all the other tested dispersion attempts in this study. According to the analysis, the samples that used Aguacyl product had the highest values of performance in the AHP, as can be seen in Table 12, the samples SM 4, SM 5, SM 6, SM 7, SM 9 and SM 10. Despite evidence of clumping in most samples with powder CNT, demonstrating that they were not fully dispersed in an aqueous medium, the study helped to understand the functioning of CNTs dispersion in various media, helping to define with a little more precision products and forms of dispersion to be used in the research. Samples with the best results with powder CNT were: SM 3 containing water, CNT and polycarboxylate additive (Tec Flow 8000 - at 1% relative to the mass of water) and SM 11 containing water, Arabic gum and CNT. It was demonstrated that the increase in sonication time of dispersion reduced the occurrence of decantation of the solutions, seen as an increasing factor for the dispersion of solutions. The study employing microscope was important because many findings based on the images obtained with this equipment revealed information that could not be obtained from observations with the naked eye. Notwithstanding, the worst dispersing results consisted in samples SM 8, with water, CNT and polycarboxylate additive (Tec Flow 8000 - at 0.5% relative to the mass of water) and SM 12 with water, powder CNT and surfactant (sodium lauryl ether sulphate), which were discarded. Comparing the results from SM 4 (Figure 9) with the ones of SM 5 (Figure 10), it was demonstrated that the introduction of polycarboxylate additive (Tec Flow 8000) caused the formation of clumps, initially non-existent, in liquid with CNTs industrially dispersed. One of the possible explanations is that electric bi polar charges, have accumulated on the surface of the particles causing the phenomenon of agglutination. This could be best explained if we knew exactly the dispersion process applied in the industrialized product. It is therefore suggested that further research be conducted to explain this fact.

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## 7. REFERENCES

American Society for Testing and Materials. *Standard Specification for Chemical Admixtures for Concrete*. ASTM C494/C494M. In: Annual book of ASTM Standards, West Conshohocken, 2013. American Society for Testing and Materials. *Standard Specification for Chemical Admixtures for Use in Producing Flowing Concrete*. ASTM C1017/C1017M. In: Annual book of ASTM Standards, West Conshohocken, 2007.

American Society for Testing and Materials. *Standard practice for applying analytical hierarchy process (AHP) to multi attribute decision analysis of investments related to buildings and buildings systems*. ASTM E 1765. In: Annual book of ASTM Standards, West Conshohocken, 2011.

Associação Brasileira de Normas Técnicas. "Chemical additives for Portland cement concrete – Requirements", (Aditivos químicos para concreto de cimento Portland – Requisitos). NBR 11768. Rio de Janeiro, 2011.

- Batiston E. R. (2012), "Carbon Nanotubes incorporation in Portland cement matrices", (Incorporação de Nanotubos de Carbono em Matrizes de Cimento Portland). Tese (Doutorado em Engenharia Civil), Universidade Federal de Santa Catarina, Florianópolis.
- Costa H. G., Correa P. S. (2010), "Construction of an AHP-based model to catch criteria weights in port-occupancy evaluation" International journal of the analytic hierarchy process; 2(1) 30-43.
- Costa H. G. (2002), "Introduction to hierarchical analysis method: multi-criteria analysis in the decision aid" (Introdução ao método de análise hierárquica: análise multicritério no auxílio à decisão), Niterói, R. J.
- Couto, G. G. (2006), "Nano nickel particles: synthesis, characterization, properties and study their use as catalysts in obtaining carbon nanotubes" (Nano partículas de níquel: síntese, caracterização, propriedades e estudo de sua utilização como catalisadores na obtenção de nano tubos de carbono), Dissertação (Mestrado em Química) Departamento de Química, Universidade Federal do Paraná, Curitiba.
- Gleize P. J. P. (2007), "Nanotechnology and construction materials" (Nanotechnologia e materiais de construção), In: ISAIA, Geraldo C. (Ed.). Materiais de construção civil e princípios de ciência e engenharia de materiais. São Paulo: IBRACON. v. 2. cap. 50, p. 1659-1685.
- Ibarra Y. S., Gaitero J. J., Erkizia E., Campillo I. (2006), "Atomic force microscopy and nanoindentation of cement pastes with nanotube dispersions", Physica Status Solidi; 203(6) 1076–1081. doi: 10.1002/pssa.200566166
- Koshio A., Yudasaka M., Zhang M., Lijima S. (2001), "A Simple Way to Chemically React Single-Wall Carbon Nanotubes with Organic Materials Using Ultrasonication", Nano Letters; 1(7) 361-363. doi: 10.1021/nl0155431
- Konsta-Gdoutos M. S., Zoi S. M., Surendra P. S. (2010), "Highly dispersed carbon nanotube reinforced cement based materials", Cement and Concrete Research; 40(7) 1052-1059. doi:10.1016/j.cemconres.2010.02.015
- Lai Y., Wang W., Wang H. (2008), "AHP and simulation-based budget determination procedure for public building construction projects. Automation in Construction", 17(5) 623-632. doi: 10.1016/j.autcon.2007.10.007
- Marcondes C. G. N. (2012), "Addition of carbon nanotubes in concrete portland cement absorption, permeability, chloride penetration and mechanical properties" (Adição de nanotubos de carbono em concretos de cimento portland absorção, permeabilidade, penetração de cloretos e propriedades mecânicas). Dissertação (Mestrado em Engenharia de Construção Civil) Departamento de Construção Civil, Universidade Federal do Paraná, Curitiba.
- Marchezetti A. L., Kaviski E., Braga M. C. B. (2011), "Application of AHP method for ranking of alternative treatment of solid waste" (Aplicação do método de AHP para a hierarquização das alternativas de tratamento de resíduos sólidos domiciliares). Ambiente Construído; 11(2) 173-187. Mattana A. J, Medeiros M. H. F., Silva N. G, Costa M. R. M. M. C. (2012), "Hierarchical analysis to choose between natural aggregate and sand rock crushing for making mortar coating" (Análise hierárquica para escolha entre agregado natural e areia de britagem de rocha para confecção de argamassas de revestimento), Ambiente Construído; 12(4) 63-79. doi: 10.1590/S1678-86212012000400006
- Makar J., Margeson J., Luh J. (2005), "Carbon nanotube / cement composites early results and potential applications", in: International Conference on Construction Materials: Performance, Innovations and Structural Implications, 3, Vancouver; p. 1-10.
- Metaxa Z. S., Seo J., Konsta-Gdoutos M. S., Hersam M. C., Shah S. P. (2012), "Highly concentrated carbon nanotube admixture for nano-fiber reinforced cementitious materials". Cement and Concrete Composites; 34(5) 612-617. doi: 10.1016/j.cemconcomp.2012.01.006
- Mehta P. K., Monteiro P. (2013) "Concrete Microstructure Properties and Materials", New York: McGraw-Hill.

Neville A. M. (1996), "Properties of Concrete", John Wiley & Sons.

Nochaiya T., Chaipanich A. (2011), "Behavior of multi-walled carbon nanotubes on the porosity and microstructure of cement-based materials", Applied Surface Science; 257(6) 1941-1945. doi:10.1016/j.apsusc.2010.09.030

Pereira E., Medeiros M. H. F., Levy S. M. (2012) "Concrete durability with recycled aggregates: an application hierarchical analysis" (Durabilidade de concretos com agregados reciclados: um aplicação de análise hierárquica), Ambiente Construído; 12(3) 125-134. doi: 10.1590/S1678-86212012000300009

Sobolkina A., Mechtcherine V., Khavrus V., Maier D., Mende M., Ritschel M., Leonhardt A. (2012) "Dispersion of carbon nanotubes and its influence on the mechanical properties of the cement marix", Cement and Concrete Composites; 34(10) 1104-1113. doi: 10.1016/j.cemconcomp.2012.07.008.

Saaty T. L. (1978) "Exploring the interface between hierarchies, multiple objectives and Fuzzy sets", Fuzzy Sets Systems"; 1 57-68.

Zarbin A. J. G. (2007) "Chemistry of nanomaterials" (Química de nano materiais), Quim. Nova; 30(6) 1469-1479.