

Selection Process and Analysis of Bibliographic Set for a Research Involving Carbon Nanotubes Dispersion Using the ProKnow-C

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Abstract - The carbon nanotubes (CNTs) dispersion in cement composites has been studied due to the contribution in strength improvements of them. Researchers are mostly studying two different kinds of dispersion: either by surfactants or by a prior dispersion of CNTs into particles of cement or other material, which involves non-aqueous environment. Aiming to elaborate an investigation in this subject, the present bibliographical review was developed to select the most aligned material for the dispersion of CNTs in the polycarboxylate and lignosulfonate surfactants and in a non-aqueous solution of isopropanol. The papers were selected and filtered, and those considered most relevant composed the Bibliographic set. In sequence, the articles were analyzed bibliometrically and systematically and determining gaps contained in this subjective. Through the analysis made, contribution opportunities were identified emphasizing the research relevance to the scientific community. Thus, the main contribution of this paper is identified gaps related to the dispersion of carbon nanotubes in cement composites and the consolidated studies to guide future research. The methodology used was Knowledge Development Process - Constructivist (ProKnow-C).

Keywords- *Bibliographic Set, Proknow-C, Carbon Nanotubes, Cement, Dispersion*

I. INTRODUCTION

Carbon nanotubes (CNTs) discovery leads to studies of the incorporation of this nanomaterial into civil construction materials. Researches have shown that CNTs incorporation allows improvement in cementitious compounds mechanical properties. However, the hydrophobic behavior of CNTs makes the dispersion of this material a challenge to the cement pastes production. For best dispersion, the CNTs are submits to treatment capable of increasing the dispersive properties. According to Filho and Fagan (2007) [1], one of the types of treatment is called functionalization and happens through non-covalent interactions (weak bonds with CNTs) and/or covalent interactions (strong interactions that generate large modifications in the properties of CNTs).

Paula (2014) [2] affirms that researchers have currently studied two methods of dispersion: (i) physical dispersion of nanotubes during composites preparation, usually by mixing in

plasticizers (surfactants) and (ii) pre-dispersion of the nanotubes in the cement particles, or other composite material, either by the in-situ synthesis of the CNTs in the particles [3] or by the dispersion in non-aqueous medium of the non-hydrated cement particles.

Before developed a work able to evaluate the mechanical performance of cement composites with the addition of MWCNTs dispersed in those two most studied forms, dispersed nanotubes in mixtures with plasticizers (lignosulfonate and polycarboxylates) and by pre-dispersion in non-aqueous isopropanol medium, it is necessary a carefully research about the currently studies made about this subjective.

The present article, therefore, consists to performing a systemic search for already accomplished researches that are to the subject and allow the greater acquisition of knowledge on the subject.

The systemic search direction was guided by the following research question: "What is the most appropriate material to compose the bibliographic set involving dispersion of carbon nanotubes in non-aqueous medium of isopropanol and mixtures of lignosulfonate and polycarboxylate plasticizers?"

The methodological framework of this research is based on the classification described by Prodanov and Freitas (2013), [4], shown in Figure 1. The research is considered basic because it aims to find out more about the dispersion of nanotubes in cement matrices, and find a solution to the hydrophobic characteristic (specific problem) of the material.

It is also considered qualitative and exploratory because it collects the information of research carried in different forms of dispersion, analyzing them and verifying their relevance for the work aimed. It is a descriptive process in which inductive data are analyzed and it allows objectives elaboration and hypotheses formulation.

It is characterized by a bibliographical research work because it is based on articles of periodicals already available.

Once the scope of the research to be carried out has been defined, the selection and analysis of the Bibliographic Set will be done by a material selection tool: ProKnow-C. According to Azevedo (2013) [5], this instrument is divided into three stages: (i) bibliographic set selection; (ii) bibliometric analysis; (iii) systemic analysis.

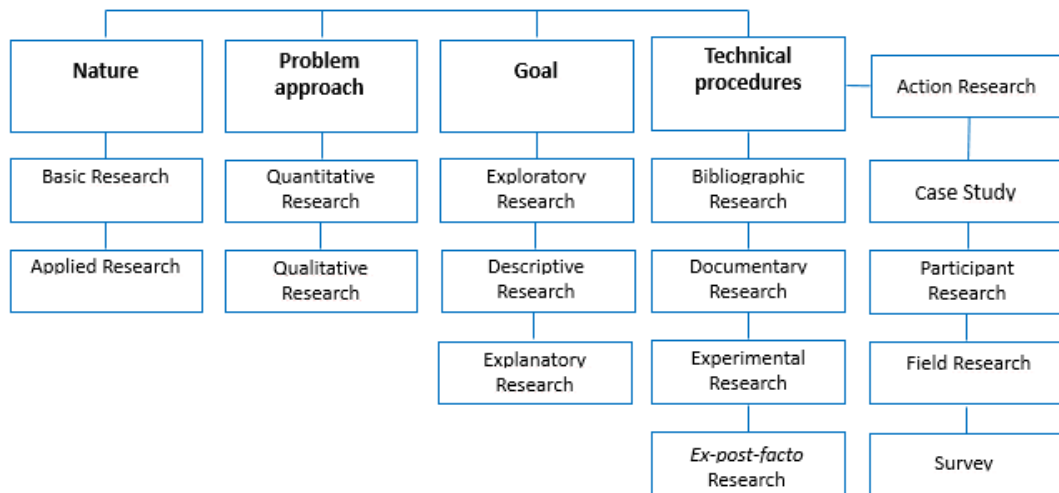


Figure 1. Research methodological framework.

II. RESEARCH DEVELOPMENT

The Research Development was carefully done following the steps mentioned above suggested by Azevedo (2013) [5].

A. Bibliographic set selection

The selection process of the bibliographic set was based on the process described by Ensslin; Ensslin; Pacheco (2012) [6].

1) Articles bank selection

According to Ensslin; Ensslin; Pacheco (2012) [6], the article selection process is composed of four phases: (i) keywords definition; (ii) databases definition; (iii) articles search in databases using the keywords; (iv) keywords adherence test.

2) Keywords definition

The considered most relevant words to the research to be developed were: Carbon nanotubes, Cement and Dispersion. These words were obtained considering the aim of the desired research, to disperse the nanotubes in the cement matrix. Since they represent the main idea of the research, those main keywords were considered.

In addition, the dispersion media used are also considered to be relevant, so Lignosulfonate (surfactant), Polycarboxylate (surfactant) and Isopropanol (non-aqueous liquid substance) can also be considered as keywords.

In this way, the following combinations will be searched in the database: (i) Carbon nanotubes, Cement, Dispersion; (ii) Carbon nanotubes, Cement, Dispersion, Lignosulfonate; (iii) Carbon nanotubes, Cement, Dispersion, Polycarboxylate; (iv) Carbon nanotubes, Cement, Dispersion, Isopropanol.

3) Databases definition

Science Direct was chosen as base for the search of the articles. This platform was chosen due to the CAPES Periodicals Portal makes available to academics free access to this database, thus allowing the largest contingent of articles search.

4) Articles search in databases using the keywords

The chosen database (Science Direct) offers advanced search option by applying filters. Using this resource, the search was limited to the Engineering area and the publications in just in periodicals. The option of temporal filters was not used, because it is a recent theme [7].

5) Keywords adherence test

The combinations determined as keywords were tested and returned the quantity of articles indicated in Table 1:

TABLE I. FILTER RESULT OF THE ARTICLES SEARCH IN THE DATABASE.

Key words combination	Filter result
Carbon nanotubes, Cement, Dispersion	147
Carbon nanotubes, Cement, Dispersion, Lignosulfonate	6
Carbon nanotubes, Cement, Dispersion, Polycarboxylate	29
Carbon nanotubes, Cement, Dispersion, Isopropanol	6

The application of the method allowed the selection of 188 articles to compose the articles database.

B. Articles bank filtering

Following the process described by Ensslin; Ensslin; Pacheco (2012) [6], the articles bank filtering follows 5 steps: (i) Redundancy; (ii) Titles of articles aligned with the research theme; (iii) Scientific recognition; (iv) Abstracts in line with the research theme; (v) Full article in line with the research topic.

1) Redundancy

The 188 selected articles were exported to the EndNote X7 reference manager. (Carlsbad and Reuters, 2015) [8]. Using the resources made available by the manager, 72 duplicate articles were identified. These articles were deleted.

2) Articles titles aligned to research topic

As a next step, it was done the reading and analysis of the titles of the articles. It allowed to identify that 96 articles were related to nanosilica, fibers, clays, graphene and geopolymers, and also related to the electrical, thermal and magnetic properties of CNTs. These articles are not aligned to the main research topic, so they were discarded. The article bank now has a total of 20 articles.

3) Scientific recognition

As a result of the previous steps, 20 articles were selected. Considering CNTs as a "current" theme and noticing that the large number of recent publications (65% of articles published in the last 5 years), the authors decided not to apply the filtering of articles by scientific recognition (number of citations). This decision was made in order to obtain a greater articles scope for selecting the bibliographic set.

4) Abstracts aligned to research topic

Reading and analyzing the abstracts of selected articles, it was allowed the identification of 10 more articles whose summary was not aligned with the research topic, so the bibliographic set consisted of 10 articles up to this stage.

5) Full article aligned to research topic

After reading all 10 articles, only 04 shows the process of dispersion of CNTs in lignosulfonate, polycarboxylate or isopropanol. In this way, the remaining six were disregarded.

At the suggestion of the supervisor of the work to be developed, 02 articles that were not included in the search process because they were indexed by other databases were analyzed and considered fully aligned with the research topic. These articles, addressing different means of dispersion, however they were considered relevant to the research.

The final validation provided 06 articles aligned to the researched topic, which compose the bibliographic set.

The keyword, Carbon nanotubes was the only word cited at least once in all Portfolio articles. Of those 06, only 03 indicate

the keywords. The terms Carbon nanotubes, Dispersion and Cement appear as keywords in 02 different articles. This indicates that the keywords that guided the search were well chosen considering the objectives proposed.

C. Bibliometric Analysis

Given the set of six articles to compose the bibliographic set, the bibliometric analysis will be developed in four stages (Ensslin, Ensslin and Pacheco, 2012) [6]: (i) Degree of relevance of the periodicals; (ii) Scientific recognition of articles; (iii) Degree of relevance of the authors; (iv) Most commonly used keywords.

1) Degree of relevance of periodicals

Of the 06 articles, each one was published in a different periodical, which indicates, that in this question, none journal presents a greater degree of relevance.

Qualis [9] assesses the quality of articles by analyzing the quality of the dissemination vehicles, i.e. scientific journals. The journals are classified as indicates in Table 2:

TABLE II. NEWSPAPERS CLASSIFICATION.

Journal	Journal Classification
Cement and Concrete Composites	A1
Journal of the American Ceramic Society	A2
Journal of Physical Chemistry C	A1
Construction and Building Materials	A2
Carbon 45	A1
Journal of Colloid and Interface Science	A2

a. A1- Impact factor equal to or greater than 3,800; A2- Impact Factor between 3,799 and 2,500; B1- Impact Factor between 2,499 and 1,300

2) Scientific articles acknowledgement

The scientific articles acknowledgement was done through the number of citations of each article. The result is shown in Table 3:

TABLE III. NEWSPAPERS CLASSIFICATION.

Article	Authors	Citations	Year of publication
The influences of admixtures on the dispersion, workability, and strength of carbon nanotube-OPC paste mixtures [10]	Collins, Frank Lambert, John Duan, Wen Hui	80	2012
Growth of cement hydration products on single-walled carbon nanotubes [11]	Makar, Jonathan M. Chan, Gordon W.	99	2009
Noncovalent functionalization of carbon nanotubes with sodium lignosulfonate and subsequent quantum dot decoration [12]	Liu, Yangqiao Gao, Lian Sun, Jing	57	2007
On the aspect ratio effect of multi-walled carbon nanotube reinforcements on the mechanical properties of cementitious nanocomposites [13]	Al-Rub, Rashid K Abu Ashour, Ahmad I Tyson, Bryan M	51	2012
Controlling the dispersion of multi-wall carbon nanotubes in aqueous surfactant solution [14]	Yu, Junrong Grossiord, Nadia Koning, Cor E. Loos, Joachim	402	2006
Comparative study of carbon nanotube dispersion using surfactants [15]	Rastogi, Richa Kaushal, Rahul Tripathi, S. K. Sharma, Amit L.Kaur, Inderpreet Bharadwaj, Lalit M.	406	2008

3) Articles content analysis

For all articles, the analysis was done according to the research lenses determined. In relation to the dispersion media, as indicated in Table 04, only 01 article uses dispersion in

nonaqueous media and the other 05 articles are dispersed in mixtures with surfactants. Of these 05 articles, 03 use the same surfactants of the desired research (lignosulfonate and polycarboxylate).

TABLE IV. ARTICLES ANALYSIS UNDER DISPERSION MEDIA CRITERION.

Article	Dispersion media
Collins; Lambert; Duan (2012) [10]	Dispersed by plasticizers Lignosulfonate and Polycarboxylates
Liu; Gao; Sun (2007) [13]	Dispersed by addition of plasticizer Lignosulfonate
Al-Rub; Ashour; Tyson (2012) [12]	Dispersed by addition of plasticizer Polycarboxylate
Yu et al. (2006) [14]	Dispersed by plasticizer Sodium Dodecyl Sulfate (SDS)
Rastogi et al. (2008) [15]	Disperse by plasticizer Triton X-100, Tween 20, Tween 80, and Sodium Dodecyl Sulfate (SDS)
Makar and Chan (2009) [11]	Disperse by addition of Isopropanol

As far as the dispersion method is concerned, according to data shown in Table 05, it can be seen that 05 articles deal with the physical dispersion of nanotubes in the preparation of

composites, which are mixed in surfactants, and only 01 article works the pre-dispersion of nanotubes in cement particles.

TABLE V. ARTICLES ANALYSIS UNDER DISPERSION METHOD CRITERION.

Article	Dispersion method
Collins; Lambert; Duan (2012) [10]	Physical dispersion of the CNTs in the preparation of the composites, by surfactants mixtures (sulphonated polynaphthalene (PNS), polycarboxylate and lignosulfonate) and air incorporation
Liu; Gao; Sun (2007) [13]	Physical dispersion of CNTs functionalized at the time of preparing the composites, by surfactants mixtures (Lignosulfonate)
Al-Rub; Ashour; Tyson (2012) [12]	Physical dispersion of the CNTs in the preparation of the composites, by surfactants mixtures (Polycarboxylates)
Yu et al. (2006) [14]	Physical dispersion of the CNTs in the preparation of the composites, by surfactants mixtures (Sodium Dodecyl Sulfide - SDS)
Rastogi et al. (2008) [15]	Physical dispersion of the CNTs in the preparation of the composites, by surfactants mixtures (Triton X-100, Tween 20, Tween 80, and Sodium Dodecyl Sulfide - SDS)
Makar and Chan (2009) [11]	Pre-dispersion of nanotubes in cement particles

Makar and Chan (2009) [11] paper presented evidence of structural reinforcement to cement composites when CNTs were pre-dispersed in non-aqueous isopropanol media. Hydration acceleration, more production of CH and greater formation of C-S-H were observed. Those facts suggest a significant degree of interfacial bonding between C-S-H and CNTs, which can be very useful for gaining composite strengths. It was not found much information about this dispersion method and it suggests a research opportunity. Thus,

it is valid to develop research to quantitatively verify the gain endorsed by Makar and Chan (2009) [11].

It is observed that none of the analyzed articles deals with comparisons between different dispersion methods. This emphasizes that the comparison of the methods is also a research opportunity in the desired work.

The concentration of materials adopted by each researcher is shown in Table 06.

TABLE VI. ARTICLES ANALYSIS UNDER MATERIALS CONCENTRATION CRITERION.

Article	Materials concentration	Ratio CNTs:Water
Collins; Lambert; Duan (2012) [10]	- Composite - Water/cement ratio: 0,4; 0,5 and 0,6 - 0,5 to 2,0% CNT (weight of cement) - 0,5 to 1,5 ml plasticizers	From 1:120 to 1:20
Liu; Gao; Sun (2007) [13]	- 0,1g CNT - 1g Lignosulfonate - 100 ml of water	1:1.000
Al-Rub; Ashour; Tyson (2012) [12]	- Composite - Water/cement ratio 0,4 - 0,025 to 0,2% CNT (weight of cement) - 0,4% Polycarboxylate (weight of cement)	From 1:1.000 to 1:200
Yu et al. (2006) [14]	- Aqueous solution - 0,01 to 0,40% CNT (weight of water) - Proportion 1,5 SDS : 1 CNT	From 1:10.000 to 1:250
Rastogi et al. (2008) [15]	- Aqueous solution with variable surfactant concentration and CNT	From 1:10.000 to 1:5.263
Makar and Chan (2009) [11]	- Non-aqueous solution of Isopropanol - Cement (water/cement ratio = 0,5) - 0 and 1% CNT (weight of cement)	1:50

Analyzing the articles according materials concentration, it is observed that 02 of them prepared cement composites with different proportions of CNTs: (i) 0,5 to 2,0% CNT(cement weight); (ii) 0,04 to 0,2% CNT (cement weight). This suggests variability in the dispersed CNTs concentration at cement particles for composites preparation. Table 6 also shows discrepancy in the ratio CNTs and water, ranging from 1:20 to 1: 10.000. This proportions variability indicates that an effective limit of dispersion in the methods studied is still not determined.

The comparison between dispersion at polycarboxylate and lignosulfonate is made in the work of Collins; Lambert; Duan (2012) [10]. The authors evidenced strength improvements (25%) in presence of polycarboxylate and 0,5% of CNTs. Differently, using lignosulfonate at the same proportion of nanotubes achieve 30,1% lower strength. The authors suggest that for better dispersion by lignosulfonate media more surfactant should be added. This suggestion is not interesting considering that it can slow down the set time.

The drop in strength observed by Collins; Lambert; Duan (2012) [10] using lignosulphonate as dispersion media may have been caused by the agglomeration of CNTs due to non-effective dispersion. A possible solution would be reduce CNTs concentration. However, this solution was not tested or considered by authors.

The results of Collins; Lambert; Duan (2012) [10] contrast the reported by Liu; Gao; Sun (2007) [13]. Liu; Gao; Sun

(2007) [13] reports in their research project the functionalization of CNTs prior to dispersion in the lignosulfonate surfactant media. The results obtained showed an effective dispersion in water, which indicates that the interactions among the CNTs were overcome by the adopted methodology. The CNTs treatment by CH₄ and HCl, hypothetically, may have been the main factor that leads to a good dispersion of nanotubes, contributing to the discrepancy in results.

The Yu et al. (2006) [14] and Rastogi et al. (2008) [15] works were developed in surfactants other than lignosulfonate and polycarboxylate, however, they presented relevant results to the desired research. Rastogi et al. (2008) [15], through tests in different types of dispersants, concluded that there is an adequate relationship between CNTs and surfactant, in which once adopted, can uniformly coat the surface of the CNTs. After the optimum ratio, dispersion capacity is reduced.

Yu et al. (2006) [14] also approach the concentration issue in order to allow homogeneous dispersion. Besides, they analyzed the variation in the sonification period. They pointed out that there is a minimum energy to obtain the maximum dispersion, and once it is reached, must not be extended to avoid CNTs damage. The results suggest that after the 90 minute period, corresponding to 100.000J of sonification energy, all CNTs practically were exfoliated, indicating a possible maximum dispersion. Data for the sonication period are shown in Table 07.

with the resistance gain and present the result both in percentage values and in microscopic images. The other articles measure the result qualitatively, according to the chemical reactions that occurred during the process and observed in microscopes. It is notice a lack of a reliable methodology to quantify CNTs dispersion. Both observations of chemical reactions by microscopes images and analysis of the composites mechanical behavior are indirect method adopted by dispersion evaluation.

TABLE VII. ARTICLES ANALYSIS UNDER SONIFICATION METHOD CRITERION.

Article	Materials concentration
Collins; Lambert; Duan (2012) [10]	12 minutes
Liu; Gao; Sun (2007) [13]	There was no sonification. The CNTs were ground by hand for 2 hours and filtered.
Al-Rub; Ashour; Tyson (2012) [12]	It was sonicated but time period was not informed
Yu et al. (2006) [14]	0 to 120 minutes
Rastogi et al. (2008) [15]	2 hours
Makar and Chan (2009) [11]	2 hours

It is noticed that 05 of the 06 articles use sonification, and according to Paula (2014) [2], it allows a covalent reaction between the materials. In the analyzed material, the minimum period was 12 minutes and the maximum of 02 hours.

Considering the results obtained by Yu et al. (2006) [14] regarding the sonification period and observing that 03 of the 05 articles kept the sonicated mixtures for 02 hours, the interval around 90 and 120 minutes can be considered a strong point to be followed.

Table 08 shows how the authors measured the results. 02 of the 06 articles in the bibliographic set present qualitative and quantitative analyzes, in which they associate the dispersion

TABLE VIII. ARTICLES ANALYSIS UNDER DISPERSION MEDIA CRITERION.

Article	Results measurement
Collins; Lambert; Duan (2012) [10]	Qualitative and quantitative analysis - Results expressed in percentage terms and microscopic images analysis
Liu; Gao; Sun (2007) [13]	Qualitative analysis - Results expressed from observations of chemical reactions of hydrated calcium silicate (CSH)
Al-Rub; Ashour; Tyson (2012) [12]	Qualitative analysis - Results expressed from observations of chemical reactions and resistance gain hypotheses
Yu et al. (2006) [14]	Qualitative and quantitative analysis - Results expressed in percentage terms and microscopic images analysis
Rastogi et al. (2008) [15]	Qualitative analysis - Results expressed from observations of chemical reactions
Makar and Chan (2009) [11]	Qualitative analysis - Results expressed from observations of the chemical reactions of hydrated calcium silicate (CSH)

Both research, Al-Rub; Ashour; Tyson (2012) and Collins; Lambert; Duan (2012), performed experiments with cementitious composites. They observed CNTs rupture when subjected to loading efforts. This means strong bonding between the cement and NTCs in presence of polycarboxylate, and embedment of nanomaterial in cement hydration products.

III. CONCLUSION

Proknow-C proposed a process for carefully select of bibliographic set, bibliometric analysis and systemic analysis that contributed to selection relevant sample to the research topic.

According to the articles analysis, there are evidences that those three media were effective to CNTs dispersion. However, due to the analysis criterion of each research, it was not possible to verify which method was the most effective, so this verification is a contribution opportunity.

In addition, in the bibliographic set, a CNTs limit dispersion at presence of lignosulfonate, polycarboxylate and isopropanol was not established, as done by Rastogi et al. (2008) for other types of surfactant. The lack of such information can be considered a research gap.

The work done by Makar and Chan (2009) suggests improvements in cement compounds strength. Thus, the quantitative verification of the strength gain through pre-dispersion in a non-aqueous media of isopropanol is also a research opportunity.

Considering the proportion of CNTs adopted, great variability was observed. The authors determined the CNTs proportion by the cement weight, and the proportion adopted varies from 0,04% to 2,0%. With 0,5% CNTs dispersed in lignosulfonate, Collins ; Lambert; Duan (2012) did not obtain positive results, which may indicate possible agglomerations. Therefore, for this surfactant, proportions higher than this content should not be verified. For polycarboxylates, the same proportion achieve positive results, which may indicate the dispersion limit is close to this value for this method. Thus, the 0,5% proportion of CNTs by cement weight dispersed in surfactants is a strong point to be considered as a base for future research.

Another strong point observed is the sonification time between 90 and 120 minutes. The conclusions reported at the analyzed material indicate that this period is adequate and, therefore, also should be adopted in future research.

Thus, through the result obtained in the analyzes, it is concluded that the present work allowed to identify gaps and recommendations to be followed. Such information will guide

the intended research, and justify its contribution to the scientific community.

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