FORM 2

THE PATENTS ACT, 1970

(39 of 1970)

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The Patent Rules, 2003

COMPLETE SPECIFICATION

(See section 10 and rule 13)

TITLE OF THE INVENTION

"A METHOD FOR PREPARING NOVEL COMPOSITION OF NANO CEMENT MORTAR BY USING HALLOYSITE NANOCLAY (HNC), AND MULTI WALLED CARBON NANOTUBES (MWCNT)"

We, applicant(s)

NAME	NATIONALITY	ADDRESS
1. Dr.N.S.Kumar	INDIAN	Professor and Head, Former director R&D, Departmant of Civil Engineering, Ghousia college of Engineering, B.M.Road Ramanagara- 562157 Karnataka, India
2. Mrs. Ghazala Anjum	INDIAN	Research Scholar, Departmant of Civil Engineering, Ghousia college of Engineering, B.M.Road Ramanagara- 562157 Karnataka, India
3. Mr. Chethan Kumar S	INDIAN	Assistant Professor, Departmant of Civil Engineering,

		Dayananda Sagar College of Engineering, Shavige Malleshwara Hills, Kumarswamy Layout, Bengaluru- 560078, Karnataka , India

The following specification particularly describes the nature of the invention and the manner in which it is performed:

FIELD OF THE INVENTION

[001] The present invention relates to the field of the role of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT). The invention more particularly relates to a method for preparing novel composition of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects on Compression, Split Tensile and Flexural strength of cement paste in sonicated and non-sonicated state.

BACKGROUND OF THE INVENTION

[002] The polyvinyl alcohol (PVA)/halloysite nanotubes (HNTs) solution are prepared with the aid of ultrasonic treatment. The composite films are prepared through casting or coagulating the PVA/HNTs solution. The coagulation process is employed to obtain a composite film without aggregation of HNTs.

[003] It is shown that the particle size and distribution of HNTs in the PVA/HNTs solution is independent of the ratio between HNTs and PVA. It is also revealed that the aggregation of HNTs takes place during the drying process of the as cast film. Compared with the film by coagulation method, the HNTs in the as cast film show less profound effect on the nucleation of the crystallization of the PVA. The crystallization temperature initially increases with HNTs loading and overloading of HNTs tend to depress the improvement in the crystallization temperature.

[004] The glass transition temperature (Tg) of the composite film decreases with HNTs loading and the aggregation process shows practically no effect on the Tg. Inclusion of HNTs greatly depresses the decomposition of the PVA backbone, while it is not effective for improving the resistance to the

abstraction of the side groups. The aggregation process at low concentration of HNTs has more significant effect on the thermal decomposition of composite films compared with that at high concentration.

[005] The conventional devices and materials are intended to its purpose and limited in scope, and using different concepts and formulation, unlike the present invention is claimable for method for preparing Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects. Therefore, it would be useful and desirable to have an improved formulation and method to meet the above-mentioned needs, therefore overcome the aforesaid problem and shortcomings.

SUMMARY OF THE PRESENT INVENTION

[006] In the view of the foregoing disadvantages inherent in the known types of methods and techniques having complex and expensive materials, are now present in the prior art. This drawback has been overcome with the usage of economical solution and method for preparing Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects, which has all the advantages of the prior art and none of the disadvantages.

[007] The present invention provides a composite electrode, which is developed with the process adopted for the testing of the specimens using i-25 Nano universal compression testing machine procured from UK is described. Specimens of size 40mmx40mmx20mm for compression testing, 40mm height and 20mm diameter for split tensile testing and specimens of size 40x20x100mm for flexure testing incorporated with 4%, 6%, 8% and 10% by weight of cement of nano materials.

[008] One of the important aspect of the present invention is to provide the specimens, which is casted with random, non uniform dispersion of nano materials in the cement. In later stages, the nanomaterial is dispersed uniformly in water by means of a sonicator. The sonication time is set for 10, 20 and 30 minutes. The specimens are then casted and subjected to experimental testing not until failure but only until threshold cracking to determine the minimum force at which the nano composite starts to fail and the results are compared with that of conventional cement paste.

[009] Furthermore, tests were conducted on i-25 Nano universal testing machine with automatic data acquisition system after 3, 7, 14 and 28 days of curing. During conduction of the test. The specimen was removed from the water and placed for surface drying for about 10 to 20 minute. The specimen was subjected to load not until failure but only until the visibility of cracks that amount to 10% breakage. EMPEROR® software from Mecmesin was used to provide commands to the machine for conduction of the test.

[010] In this respect, before explaining at least one object of the invention in detail, it is to be understood that the invention is not limited in its application to the details of set of rules and to the arrangements of the various models set forth in the following description or illustrated in the working examples. The invention is capable of other objects and of being practiced and carried out in various ways, according to the need of that industry. Also, it is to be understood that the phraseology and terminology employed herein are for the purpose of description and should not be regarded as limiting.

[011] These together with other objects of the invention, along with the various features of novelty which characterize the invention, are pointed out with

particularity in the disclosure. For a better understanding of the invention, its operating advantages and the specific objects attained by its uses, reference should be made to the accompanying drawings and descriptive matter in which there are illustrated preferred embodiments of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

[012] The invention will be better understood and objects other than those set forth above will become apparent when consideration is given to the following detailed description thereof. Such description makes reference to the annexed drawings wherein:

[013] FIG. 1-2, illustrates Carbon Nanotubes & Halloysite Nanoclay, in accordance with an embodiment of the present invention; and

[014] FIG. 3-14 depicts various results in graphical representation forms to implement methods of the present disclosure.

DETAILED DESCRIPTION OF THE INVENTION

[015] While the present invention is described herein by way of example using embodiments and as used throughout this description, the word "may" is used in a permissive sense (i.e. meaning having the potential to), rather than the mandatory sense, (i.e. meaning must). Further, the words "a" or "an" mean "at least one" and the word “plurality” means “one or more” unless otherwise mentioned. Furthermore, the terminology and phraseology used herein is solely used for descriptive purposes and should not be construed as limiting in scope. Language such as "including," "comprising," "having," "containing," or "involving," and variations thereof, is intended to be broad and encompass the subject matter listed thereafter, equivalents, and additional subject matter

not recited, and is not intended to exclude other additives, components, integers or steps. Likewise, the term "comprising" is considered synonymous with the terms "including" or "containing" for applicable legal purposes. Any discussion of documents, acts, materials, devices, articles and the like is included in the specification solely for the purpose of providing a context for the present invention. It is not suggested or represented that any or all of these matters form part of the prior art base or are common general knowledge in the field relevant to the present invention.

[016] In this disclosure, whenever a composition or an element or a group of elements is preceded with the transitional phrase “comprising”, it is understood that we also contemplate the same composition, element or group of elements with transitional phrases “consisting of”, “consisting”, “selected from the group of consisting of, “including”, or “is” preceding the recitation of the composition, element or group of elements and vice versa.

[017] The present invention is described hereinafter by various embodiments with reference to the accompanying drawings, wherein reference numerals used in the accompanying drawing correspond to the like elements throughout the description. This invention may, however, be embodied in many different forms and should not be construed as limited to the embodiment set forth herein. Rather, the embodiment is provided so that this disclosure will be thorough and complete and will fully convey the scope of the invention to those skilled in the art. In the following detailed description, numeric values and ranges are provided for various aspects of the implementations described. These values and ranges are to be treated as examples only and are not intended to limit the scope of the claims. In addition, a number of materials are

identified as suitable for various facets of the implementations. These materials are to be treated as exemplary and are not intended to limit the scope of the invention.

[018] The present invention discloses a method for preparing novel composition of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects. The method is comprised of steps, but not limited to, with enhanced synergistic effects on Compression, Split Tensile and Flexural strength of cement paste in sonicated and non-sonicated state: providing, cementitious composites havin high compressive strength and modulus of elasticity, and relatively low tensile strength, toughness and ductility; and a plurality of reinforcing agents are used with Halloysite Nano Clay.

Best mode & enablement of the present invention to enable a person ordinarily skilled in the art

[019] In one of the exemplary embodiment of the present invention, Cementitious composites have high compressive strength and modulus of elasticity, but relatively low tensile strength, toughness and ductility. In order to compensate for that, additional reinforcing agents are used to hold the cement matrix in a much stronger way as compared to conventional calcium silicate hydrate gel. Because of CNT's incredible strength and binding properties, they are considered ideal reinforcing agents. Another material that has been used in the experimental study is Halloysite Nano Clay. Halloysites are naturally occurring eco-friendly nanotubes with low cost. They have higher aspect ratio and easy dispersability in polymer matrix. In this paper, the experimental process adopted for the testing of the specimens using i-25

Nano universal compression testing machine procured from UK is described. Specimens of size 40mmx40mmx20mm for compression testing, 40mm height and 20mm diameter for split tensile testing and specimens of size 40x20x100mm for flexure testing incorporated with 4%, 6%, 8% and 10% by weight of cement of nano materials. The specimens are casted with random, non-uniform dispersion of nano materials in the cement. In later stages, the nanomaterial is dispersed uniformly in water by means of a sonicator. The sonication time is set for 10, 20 and 30 minutes. The specimens are then casted and subjected to experimental testing not until failure but only until threshold cracking to determine the minimum force at which the nano composite starts to fail and the results are compared with that of conventional cement paste.

Methodology

[020] Specimens of size 40mmx40mmx20mm for compression testing, 40mm height and 20mm diameter for split tensile testing and specimens of size 40x20x100mm for flexure testing are casted with OPC 53 grade cement and sand passing through 1.18 mm IS sieve. CNT and HNC was added into the specimen in dosages of 4 wt% [CNT(4), HNC(4)], 6 wt% [CNT(6), HNC(6)], 8 wt% [CNT(8), HNC(8)] and 10 wt% [CNT(10), HNC(10)] of the cement. W/C ratio was fixed at 0.45. Moulds for the specimen were obtained by cutting out the specimen size from Styrofoam boards. Experiment was carried out with 3 specimens for each of the composition. Specimens were casted in 2 cases

a. Non- sonicated specimens

b. Sonicated Specimens

[021] In case of Non- sonicated specimens. Cement was mixed with sand in the ratio 1:2 along with 0.45 W/C ratio. Firstly a dry mix was prepared. CNT's and HNC were added to the dry mix itself. Then water was added to the dry mixture. Since quantity was water is very small for the required specimen dimensions. Care was taken to ensure the water did not spill out of the dry mix during the addition. The wet mix was then placed into the moulds in 3 layers compacting each layer 25 times. Since the size of specimen is small, glass rod was used for providing compaction. The moulds were then placed for air drying for 24 hours. The samples were separated from the moulds and placed into curing boxes. In case of Sonicated samples, the required amount of nanomaterial was added to the water. Then, the container was kept in the sonicator for 10, 20 and 30 minutes at room temperature. Once the time passes, the sonicator switches off automatically. The tray is taken out and water is added to the cement sand dry mix. The procedure forward is the same as that done with non sonicated samples.

[022] Tests were conducted on i-25 Nano universal testing machine with automatic data acquisition system after 3, 7, 14 and 28 days of curing. During conduction of the test. The specimen was removed from the water and placed for surface drying for about 10 to 20 minute. The specimen was subjected to load not until failure but only until the visibility of cracks that amount to 10% breakage. EMPEROR® software from Mecmesin was used to provide commands to the machine for conduction of the test. The procedure is as follows:

1. Upon opening the software, go to preferences and set the required output units of load and deflection. One can also opt for stress as the output unit.

2. Insert the test program into the software telling the machine as to how it should go about the procedure of testing. The program adopted for the experiment is as follows:

a. Clear data

b. Zero value (of load and displacement)

c. Run @ 100mm/min till the load of 0.5N

d. Clear Data

e. Zero value (of load and displacement)

f. Hold at displacement of 0 mm for 5 seconds.

g. Run @ 5mm/min until 900 N or 2 mm displacement or 10% breakage.

[023] In accordance with another embodiment of the present invention, the clear data command is used to erase any data of the previous conducted test. Zero value of load and displacement is used to set the datum of the machine value to zero with respect to load and displacement irrespective of the position of the machine. The specimen is placed and the machine is started. The load cell is lowed to the specimen at the rate of 100 mm/min until the bottom of the load cell just touches the surface of the specimen. The load cell halts at this position for 5 seconds as per the HOLD command. After this the original testing takes place when load is applied to the specimen at the rate of 1 mm/min until the load value of the load cell reaches 900N or the top displacement due to buckling is at the 10mm or until there is 10% breakage of the specimen, whichever comes first. After the test, the load cell automatically reverts back to its zero position.

[024] In the software, a graph of load v/s deflection is plotted as the test is being conducted. Calculations of maximum load, maximum displacement, average value, sum etc can be done in the software itself. Once the test is completed, the software automatically displays the value of maximum load at which the specimen undergoes 10% breakage, hence the stress and other data obtained is minute in nature.

[025] The above-mentioned invention is aimed to forming the specimens of, but not limited to, size 40mmx40mmx20mm for compression testing, 40mm height and 20mm diameter for split tensile testing and specimens of size 40x20x100mm for flexure testing incorporated with 4%, 6%, 8% and 10% by weight of cement of nano materials. Further, the specimens are casted with random, non-uniform dispersion of nano materials in the cement.

[026] Other embodiments of the invention will be apparent to those skilled in the art from a consideration of the specification or practice of the invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with the true scope and spirit of the invention being indicated by the following claims.

[027] The aforesaid features makes the present invention novel and inventive to achieve reliability and performance overcoming the drawbacks from the pre-existing process and methods is invented. It is to be understood that the above description is intended to be illustrative, and not restrictive. For example, the above-discussed embodiments may be used in combination with each other. Many other embodiments will be apparent to those of skill in the art upon reviewing the above description.

[028] The foregoing examples are but a few of the total possible parameters and lab conditions which may be developed using the basic ingredients and it should be understood the recitation of these compositions is not intended to limit in any way the scope of this invention.

[029] The benefits and advantages which may be provided by the present invention have been described above with regard to specific embodiments. These benefits and advantages, and any elements or limitations that may cause them to occur or to become more pronounced are not to be construed as critical, required, or essential features of any or all of the embodiments.

[030] While the present invention has been described with reference to particular embodiments, it should be understood that the embodiments are illustrative and that the scope of the invention is not limited to these embodiments. Many variations, modifications, additions and improvements to the embodiments described above are possible. It is contemplated that these variations, modifications, additions and improvements fall within the scope of the invention.

We Claim:

1. A method for preparing novel composition of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects on Compression, Split Tensile and Flexural strength of cement paste in sonicated and non-sonicated state, comprising the steps of:

providing, cementitious composites havin high compressive strength and modulus of elasticity, and relatively low tensile strength, toughness and ductility; and

a plurality of reinforcing agents are used with Halloysite Nano Clay.

2. The method as claimed in claim **1**, wherein the specimens of size 40mmx40mmx20mm for compression testing, 40mm height and 20mm diameter for split tensile testing and specimens of size 40x20x100mm for flexure testing incorporated with 4%, 6%, 8% and 10% by weight of cement of nano materials.

3. The method as claimed in claim **1**, wherein the specimens are casted with random, non-uniform dispersion of nano materials in the cement.

4. The method as claimed in claim **1**, wherein in later stages, the nanomaterial is dispersed uniformly in water by means of a sonicator.

5. The method as claimed in claim **3**, wherein the sonication time is set for 10, 20 and 30 minutes and the specimens are then casted and subjected to

experimental testing not until failure but only until threshold cracking to determine the minimum force at which the nano composite starts to fail and the results are compared with that of conventional cement paste.

6. The method as claimed in claim **1**, wherein the CNT and HNC is added into the specimen in dosages of 4 wt% [CNT(4), HNC(4)], 6 wt% [CNT(6), HNC(6)], 8 wt% [CNT(8), HNC(8)] and 10 wt% [CNT(10), HNC(10)] of the cement.

7. The method as claimed in claim **1**, wherein W/C ratio is fixed at 0.45, and further moulds for the specimen were obtained by cutting out the specimen size from Styrofoam boards.

8. The method as claimed in claim **1**, wherein the experiment is carried out with 3 specimens for each of the composition, and casted in 2 cases includes i. Non-sonicated specimens, ii. Sonicated Specimens.

9. The method as claimed in claim **1**, wherein in case of Non- sonicated specimens, the cement was mixed with sand in the ratio 1:2 along with 0.45 W/C ratio; firstly a dry mix was prepared. CNT's and HNC were added to the dry mix itself; then water was added to the dry mixture; Since quantity was water is very small for the required specimen dimensions; Care is taken to ensure the water without spill out of the dry mix during the addition; the wet mix was then placed into the moulds in 3 layers compacting each layer 25 times; Since the size of specimen is small, glass rod was used for providing compaction; the

moulds are then placed for air drying for 24 hours, and further, the samples were separated from the moulds and placed into curing boxes.

10. The method as claimed in claim **1**, wherein in case of Sonicated samples, the required amount of nanomaterial is added to the water; then, the container was kept in the sonicator for 10, 20 and 30 minutes at room temperature; once the time passes, the sonicator switches off automatically; the tray is taken out and water is added to the cement sand dry mix, and the procedure forward is the same as that done with non sonicated samples.

Dated this 17th day of August, 2021

Applicant(s)

Dr.N.S.Kumar et. al.

ABSTRACT

A METHOD FOR PREPARING NOVEL COMPOSITION OF NANO CEMENT MORTAR BY USING HALLOYSITE NANOCLAY (HNC), AND MULTI WALLED CARBON NANOTUBES (MWCNT)

[031] The present invention discloses a novel composition of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) and preparation method thereof. The method for preparing novel composition of Halloysite nanoclay (HNC), and Multi Walled Carbon Nanotubes (MWCNT) with enhanced synergistic effects on Compression, Split Tensile and Flexural strength of cement paste in sonicated and non-sonicated state, comprising the steps of, but not limited to: providing, cementitious composites havin high compressive strength and modulus of elasticity, and relatively low tensile strength, toughness and ductility; and a plurality of reinforcing agents are used with Halloysite Nano Clay.

Accompanied Drawings [FIG. 1-2]

Dated this 17th day of August, 2021

Applicant(s)

Dr.N.S.Kumar et. al.

FIG. 1

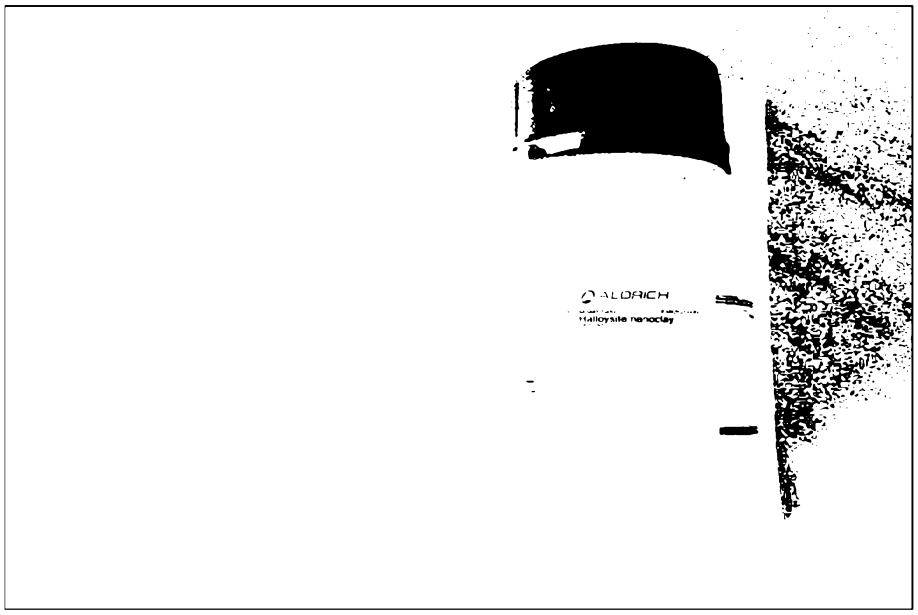
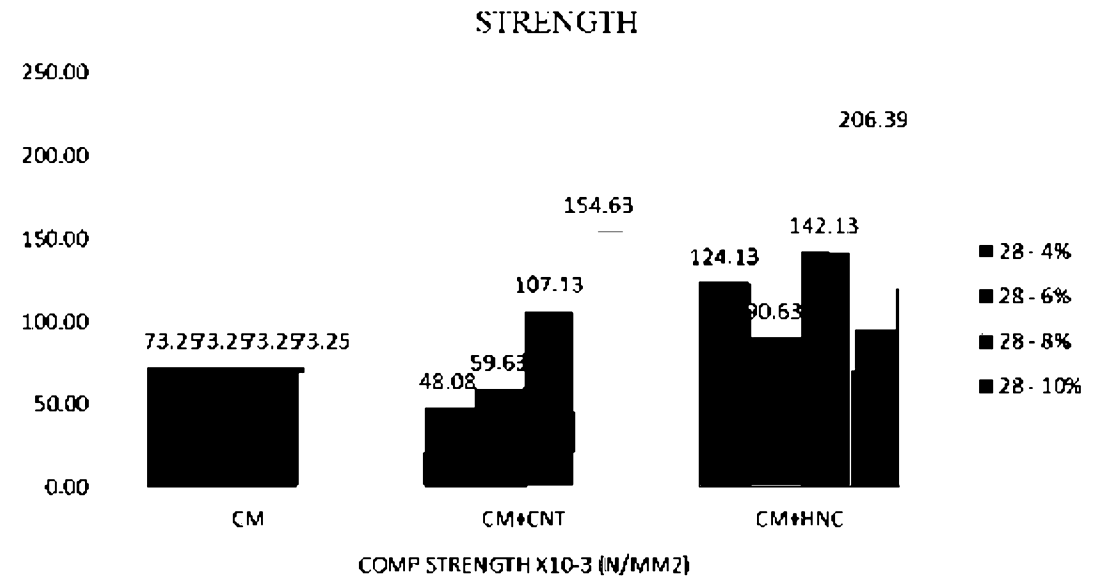
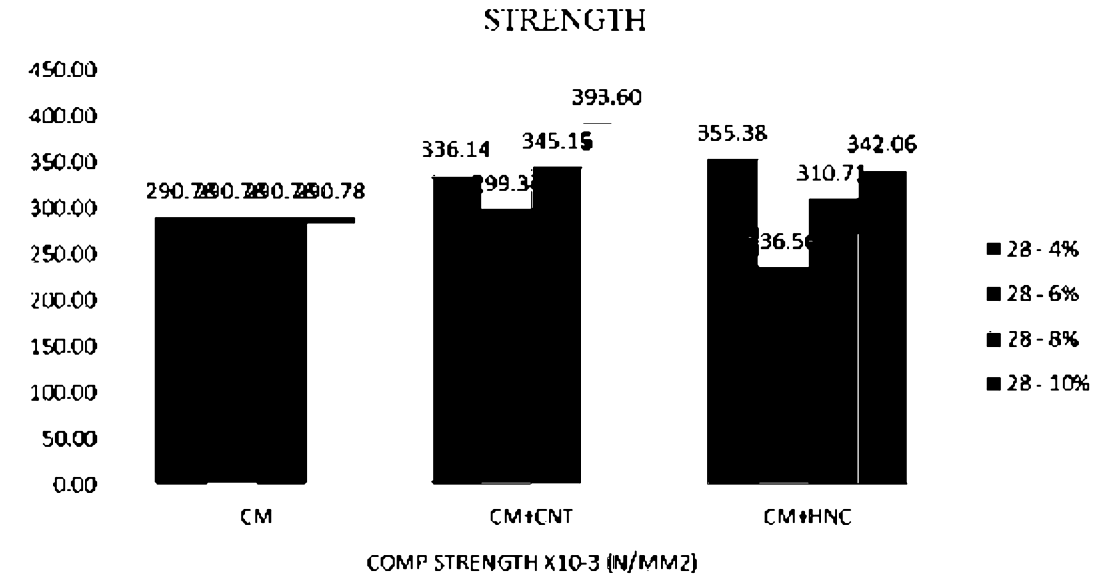


FIG. 2



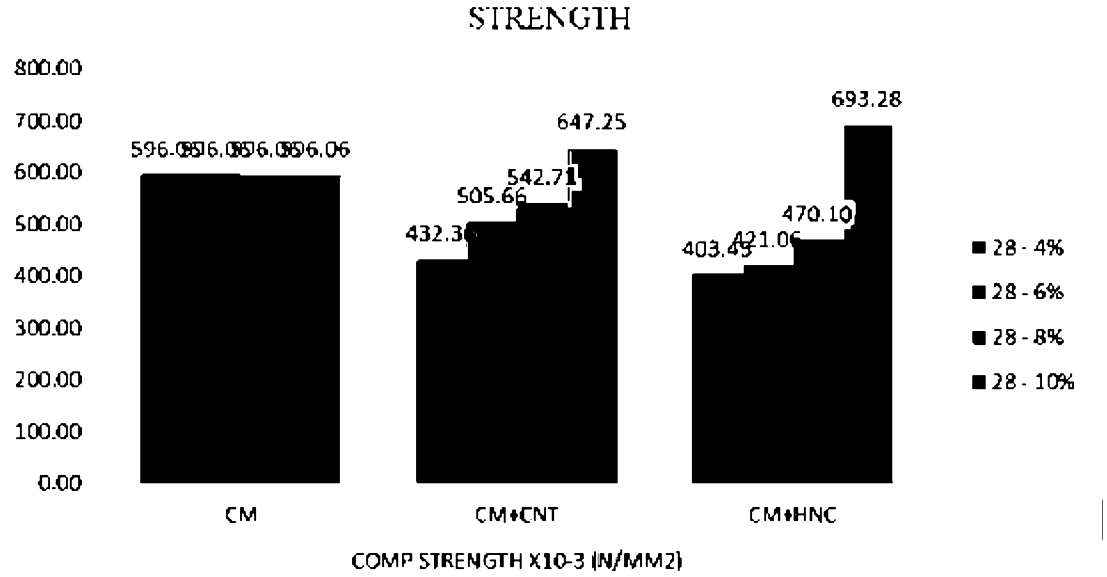
Graph representing 28 day strength for non sonicated specimens

FIG. 3



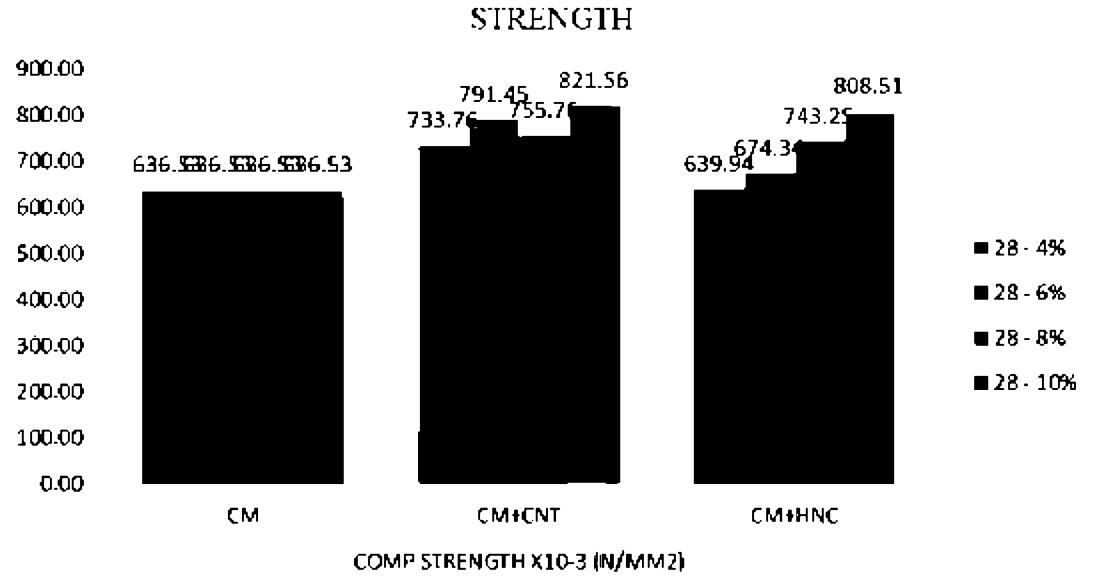
Graph representing 28 day strength for sonicated specimens (Sonication time- 10 min)

FIG. 4



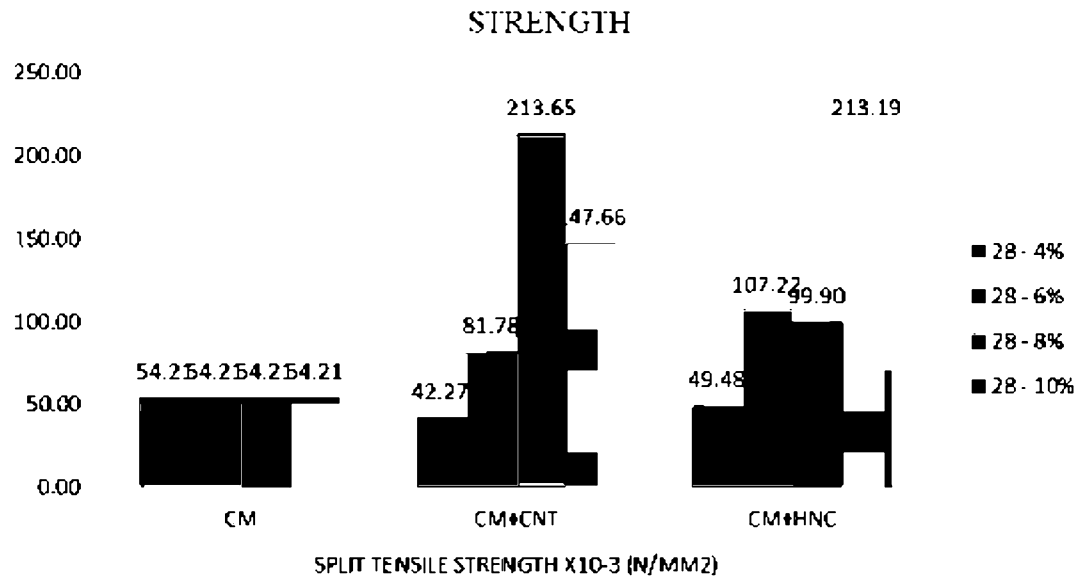
Graph representing 28 day strength for sonicated specimens (Sonication time- 20 min)

FIG. 5



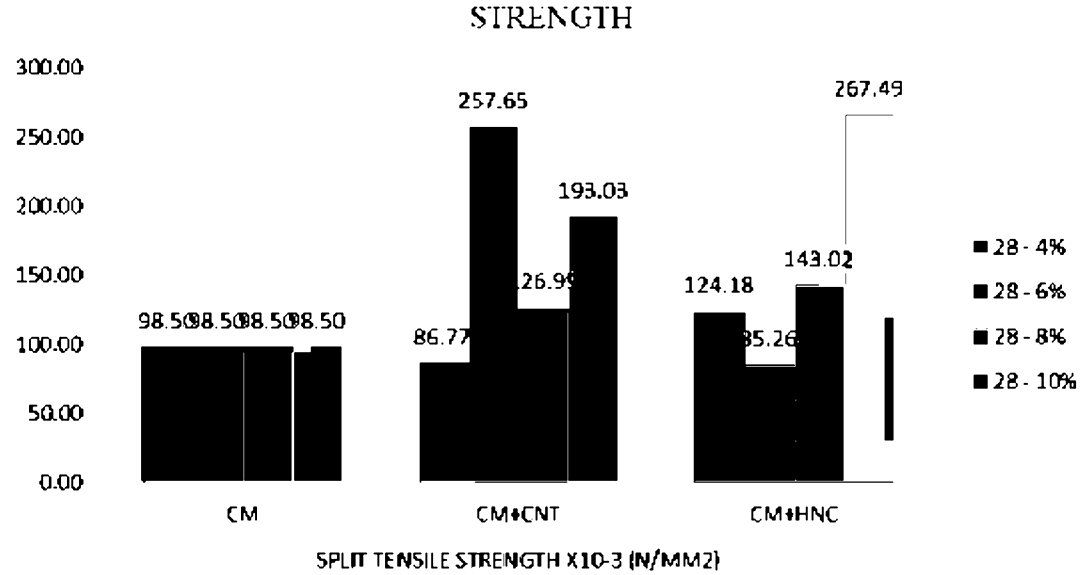
Graph representing 28 day strength for sonicated specimens (Sonication time- 30 min)

FIG. 6



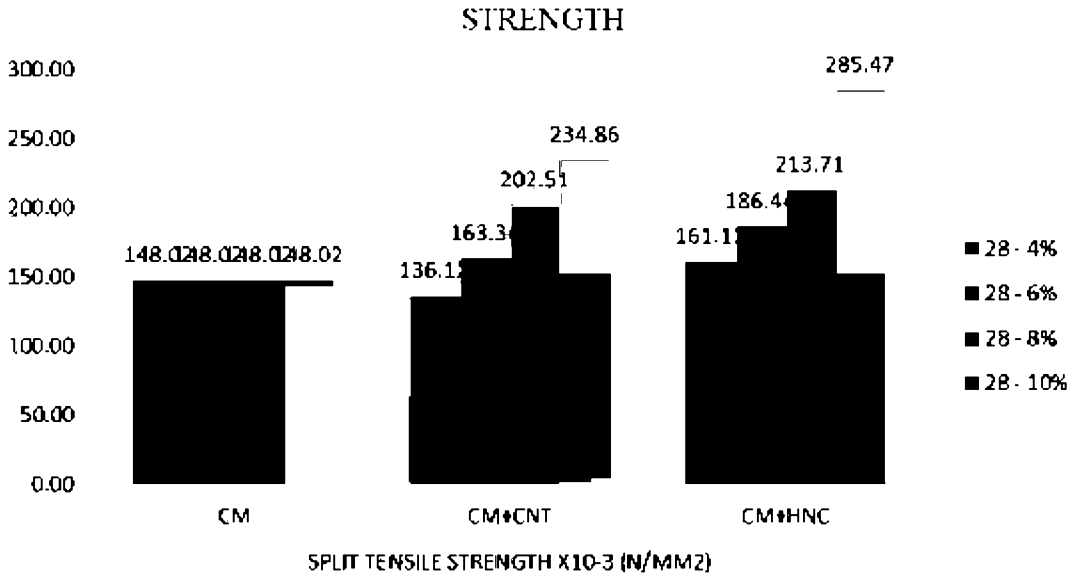
Graph representing 28 day strength for non sonicated specimens

FIG. 7



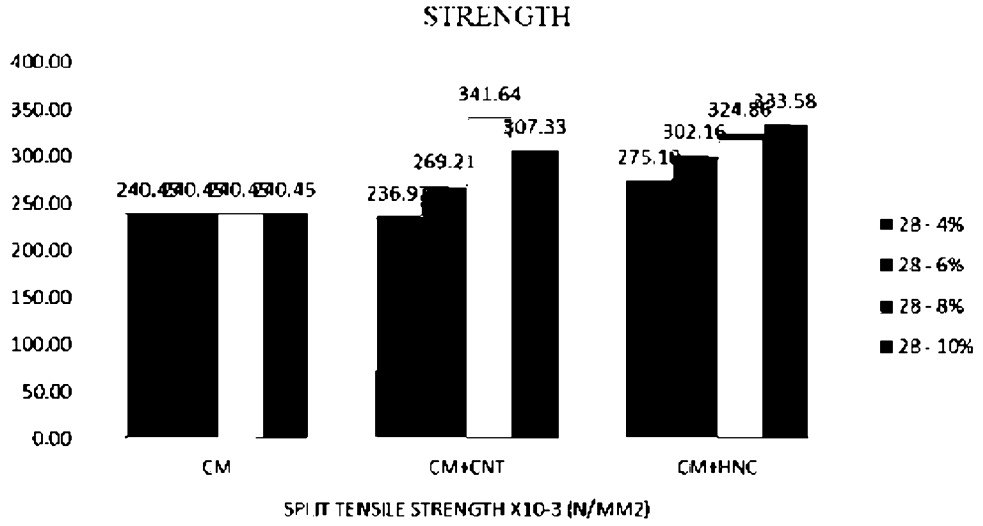
Graph representing 28 day strength for sonicated specimens (Sonication time- 10 min)

FIG. 8



Graph representing 28 day strength for sonicated specimens (Sonication time- 20 min)

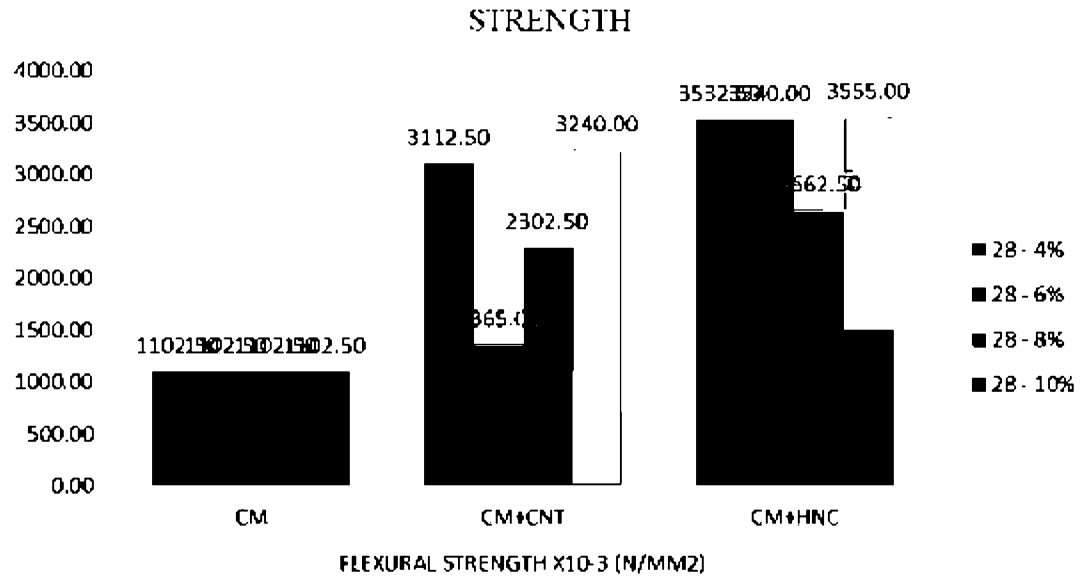
FIG. 9



Graph representing 28 day strength for sonicated specimens (Sonication time- 30 min)

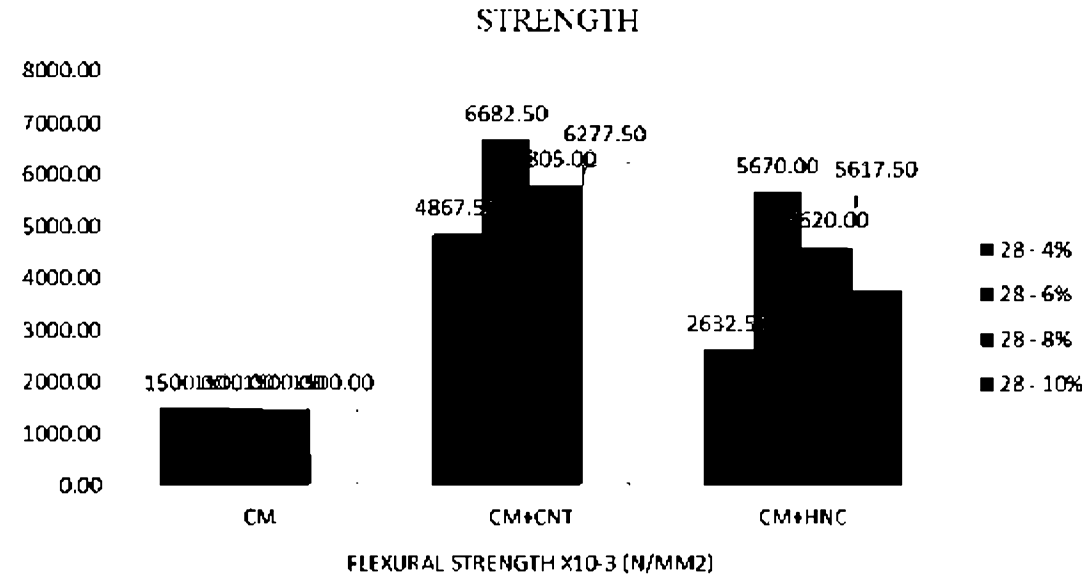
Flexural Testing

FIG. 10



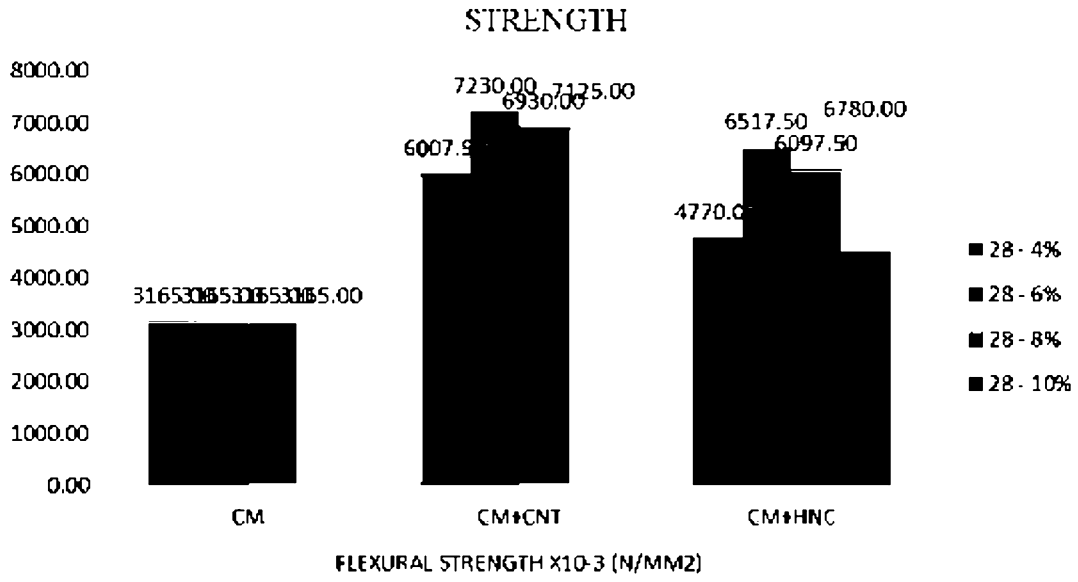
Graph representing 28 day strength for non sonicated specimens

FIG. 11



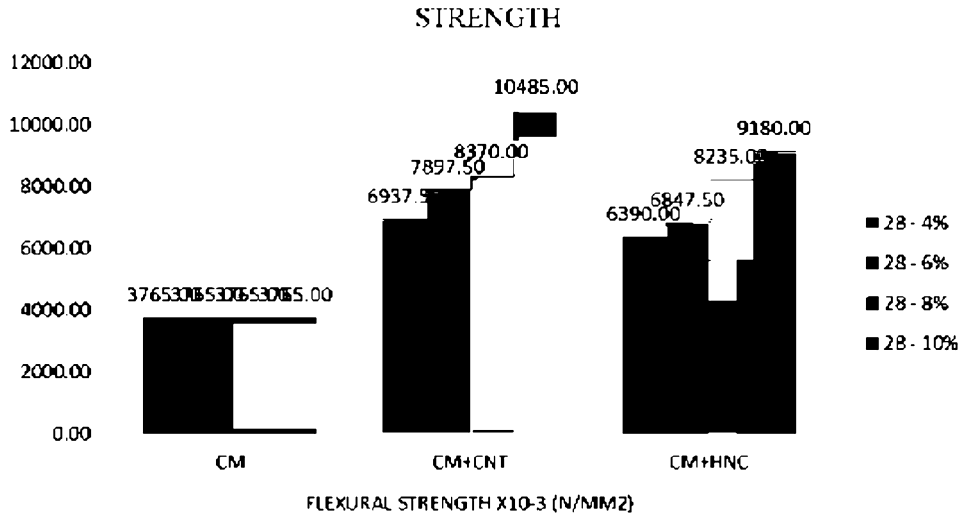
Graph representing 28 day strength for sonicated specimens (Sonication time- 10 min)

FIG. 12



Graph representing 28 day strength for sonicated specimens (Sonication time- 20 min)

FIG. 13



Graph representing 28 day strength for sonicated specimens (Sonication time- 30 min)

FIG. 14

Dated this 17th day of August, 2021