ORIGINAL RESEARCH PAPER

Hard water treatment with synthesized carbon nanoparticles of Phyllanthus emblica

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ABSTRACT

Water is, indisputably, the most vital component of life on earth. Water is a life elixir and its hardness is defined by the high concentration of magnesium, calcium, lead, chromium, iron, and mercury. The hardness of water limits its domestic and industrial usage severely. Therefore it is essential to suggest a simple, low-cost, and robust method for hard water treatment by evaluating the results in terms of Physicochemical parameters. In this paper, an efficient approach for hard water treatment by using synthesized carbon nanoparticles (C NPs) of Phyllanthus Emblica wood barks. The water samples are collected from the towns and villages located in Virudhunagar and Tuticorin District. For hardness treatment, in this work the physicochemical parameters considered are pH, TDS, dissolved Oxygen, Ca, Mg, Chloride, Alkalinity, and hardness levels. The experimental analysis, cleared that the proposed carbon nanoparticles synthesized from Phyllanthus Emblica wood barks are a very efficient and cost-effective solution.

Keywords: Physico-chemical properties, Carbon Nanoparticles, Phyllanthus Emblica, XRD analysis, hard water treatment.

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INTRODUCTION

Toxic symptoms of taking too much supplemental magnesium include a drop in blood pressure, an abnormal cardiac rhythm, muscle weakness, difficulty breathing, and deterioration of kidney function. Hard water is traditionally softened by chemical methods such as lime soda treatment or ion exchange methods. The treatment of lime soda results in a large amount of precipitate, particularly magnesium hydroxide and calcium carbonate. Furthermore, backwashing is required for ion exchange resins, and the recharged water (brine) contains a significant amount of salt. Both of these by-products can be harmful to the environment if not properly disposed of. However, these methods are all harmful to the environment in some way. As a result, there is a need for a simple

environment-friendly, and cost-effective method to remove hardness-producing salts from water.

Among the various physical methods of water purification such as boiling, filtration, adsorption, and photocatalytic process, adsorption has consistently been found to be economical and suitable due to its effective treatment and cost efficiency.

The nature of hard water can be altered by vetiver root (55.93%), Indian gooseberry bark (42.14%), lemon peel (42%), and peanut husk (41.14%). The reduction of total hardness was reported in earlier studies [1]. Peanut husk showed the lowest % decrease in total hardness. Thus, vetiver root seems to be more effective in reducing the total hardness of hard water than any other plant material. And also peanut husk is capable of retaining its adsorptive property more than other

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plant materials. The Indian gooseberry through reduces total hardness in both loading (1002ppm) and reloading (810 ppm) of hard water samples, also proved that it can be used only for a few recycles when compared to other plant parts as it has a low percentage of decrease in hard water samples reloading (19.16%) than sample first loading (28.43%). All the plant materials showed a decrease in total hardness reduction between the first and second sample loading.

Phyllanthus Emblica is a deciduous tree that is also known as Indian Gooseberry or amla. It is also known as nellikani in Tamil. It is a member of the Phyllanthaceae family. It is widely grown in many agricultural regions throughout India. Water's physicochemical properties were altered by Phyllanthus Emblica wood [2]. The chelation property of Emblica Officinalis wood may be responsible for the reduction of magnesium levels in the water. Because magnesium salts are more soluble than calcium salts, they increase the hardness of water, impart an unpleasant taste, and may have laxative effects when consumed in high concentrations [3]. In this study, Emblica Officinalis wood was chosen as a natural product of biological origin to reduce hardness, as it is commonly used for drinking water treatment in rural areas of India and many African countries. In the Indian peninsula, the wood of Amla (Phyllanthus Emblica) is used to clear small rain ponds in order to obtain safe and healthy drinking water [4,5]. This is a significantly less expensive method of obtaining contaminated water fit for human consumption.

As a result, the purpose of this study is to evaluate the quality of hardness of bore-well water sources in and around the Virudhunagar district of Tamil Nadu, as well as the water hardness removal property of Amla wood.

Recently, [6] wrote a review about the development of carbon-based functional nanomaterials describing all versatility, functionalities, and potential application in diverse fields. In this review, the authors demonstrated that carbon-based nanomaterials exhibit distinct physical and chemical properties such as chemical stability, and good thermal and electric conductivity, which could improve optical properties, besides displaying great potential for applications in material preparation, environmental science, energy storage, pharmaceutical analyses, and medical science. All these factors made carbon nanomaterials attract great attention since they were first reported.

So for the removal of hardness from water samples, Emblica Officinalis barks are converted into nanoparticles to evaluate the efficiency of the hardness removal process. This would be a better alternative, environmentally friendly, and also a low-cost solution, to make available soft water for human consumption and industrial purposes.

As a result, the purpose of this study is to evaluate the quality of hardness of bore-well water sources in and around the Virudhunagar district of Tamil Nadu, as well as the water hardness removal property of Amla wood.

MATERIALS AND METHODS

Synthesis of Carbon Nanoparticles from Phyllanthus Emblica Bark

Fresh and high-quality Phyllanthus Emblica barks were collected. The barks are cleaned with distilled water and the coat from the barks is removed and cut into small pieces. These pieces are dried for 4 hours in a hot air oven at 250°C, then ground to a fine powder with a mortar and pestle. Finally, carbon nanoparticles (C NPs) are synthesized from Phyllanthus Emblica barks are stored in an airtight container for further analysis and application.

Morphological Study with Scanning Electron Microscope

To characterize the surface morphology of the synthesized carbon NPs, the Scanning Electron Microscope (SEM)-EVO18 (CARL ZEISS) was used. SEM is a popular method for scanning the surface with a focused electron beam to create high-resolution imaging of the surfaces. This SEM can be employed to characterize the nano-scale materials too. The collected carbon NP samples were placed in an evacuated chamber and scanned in a controlled pattern by interacting with an electron beam. As a result of the interaction of this electron beam with the carbon NPs, it produces the SEM images of carbon NPs.

Structural Property with X-ray diffraction

X-ray diffraction (XRD) is one of the most extensively used techniques for the characterization of NPs. Most commonly, X-ray diffractometer D8 Advance ECO (Bruker) is used to study the grain size of carbon NPs. This XRD is capable of distinguishing the crystalline structure based on the nature of the phase, lattice parameters, and

crystalline grain size. The nanocrystalline size of the grain is evaluated by using the Scherrer equation [14-16] by broadening the most intense peak of an XRD measurement for a specific sample. The crystallite size here corresponds to the size of the grains of carbon NPs for the respective diffraction peak.

Hard water samples preparation for treatment

Rainwater samples (RW_i) and bore-well water samples (BW_j) were collected from Viruthunagar and Tuticorin districts in Tamil Nadu and these are sequenced as RW₁ to RW₃ and BW₁ to BW₆. These samples are carefully collected in sterilized and phosphate-free bottles.

Hard water treatment and analysis

The collected water samples were analyzed on various physicochemical (Magnesium, Calcium, Chloride, hardness, pH, TDS, alkalinity, and dissolved Oxygen) [7-10].

Procedure for water analysis

The procedure for water analysis has followed "Standard Methods of Analysis of Water and Wastewater13" (APHA) [11]. All the measurements were carried out in the vicinity of temperature 30°C.

Hardness

Hardness in water is due to the presence of dissolved salts of calcium and Magnesium. The estimation of hardness is based on complex metric titration. The hardness of Water is determined by titrating with a standard solution of ethylene diamine tetra acetic acid (EDTA) which is a complexing agent. Since EDTA is insoluble in water. The disodium salt of EDTA is taken for this experiment. EDTA can form four or six coordination bonds with a metal ion.

Total hardness

Total hardness is due to the presence of bicarbonates, chlorides, and sulfates of calcium and magnesium ions. The total hardness of water is estimated by titrating the water sample against EDTA using Eriochrome Black-T (EBT) indicator. Initially, EBT forms a weak wine-red colored complex with Ca²⁺/Mg²⁺ ions present in the hard water. In addition to the EDTA solution, Ca²⁺/Mg²⁺ ions preferably form a stable EDTA - Ca²⁺/Mg²⁺ complex with EDTA leaving the free EBT indicator

in the solution which is steel blue in the presence of ammonia buffer.

The concentration of calcium and Magnesium ions

The concentration of calcium and magnesium ions is determined by using the EDTA method

The concentration of chloride ions

Chloride was determined by the argentometric method. 1.0ml of 5% potassium chromate solution was added to 20.0ml of the sample and titrated with standard 0.014N ${\rm AgNO_3}$ solution till the color changed to reddish brown.

Total dissolved solids

A clear dry glass beaker of 100 mL capacity (which was kept at 100°C in an oven for 1 hour) and put an appropriate identification mark on it. The weight of the Beaker was noted (w_1) . 50ml of the water sample was heated for 2 hours. When the water has evaporated, the beaker was cooled and weighed. The weight of the beaker with precipitate (dissolved solids) was noted (w_2) .

Total dissolved solids = w_2 - w_1 / water sample $\times 10^6$ ppm

Dissolved oxygen

200ml of the water sample was taken in 300ml bottle, 1ml of 0.414M MnSO₄ solution was added followed by 1ml of Winkler reagent. The solution was mixed thoroughly. When the precipitate had settled sufficiently 1ml conc.H₂SO₄ was added. The bottle was re-stoppered and the contents were mixed well. This solution was titrated with 0.025M thiosulphate solution to pale straw color. A few drops of the starch solution were added and titration was continued up to the first disappearance of the blue color.

pН

pH - 009 (I)A pen-type pH meter of 0.01 readability was used for the measurement of pH.

Alkalinity

Take 20ml of water sample in a conical flask. Now add 3 to 4 drops of phenolphthalein indicator, the solution turns pink, and then titrate the solution with 0.1N HCl till the pink color disappears. Now, add 3 to 4 drops of methyl orange indicator to the same solution and continue the titration until the yellow color of the solution turns pink.

Calculation:



Phenolphthalein alkalinity

 $X_{_}A \times N_{HCL} \times 1000 \times 50$ /volume of sample Methyl orange alkalinity

 $Y_B \times N_{HCL} \times 1000 \times 50$ /volume of sample Total alkalinity

 $Z_{=}(A+B) \times N_{HCL} \times 1000 \times 50$ /volume of sample Here, A = Volume of HCl concerned when A phenolphthalein indicator is used.

B= Volume of HCl concerned when Methyl an orange indicator is used.

The procedure for water analysis has followed "Standard Methods of Analysis of Water and Wastewater13" (APHA) [11]. All the measurements were carried out in the vicinity of temperature 30°C.

During this analysis, on Day 0 after the sample was collected, for each of the water samples, all the above-mentioned physicochemical parameters were measured [12, 14]. Then in 2L of the water sample, 20mg of the synthesized nanoparticles were added and the mixture is shaken well with a help of a mechanical shaker. the water treatment analysis was carried out for 5 consecutive days and the data were noted down.

RESULTS AND DISCUSSION

SEM Analysis of carbon NPs

Based on the morphological studies of the synthesized carbon NPs with SEM analysis, the resultant SEM image was obtained through the interaction of an electron beam with carbon NPs. The SEM images of synthesized Carbon NPs were shown in Fig. 2.

The SEM results show that there is no uniformity in the size and shape of the nanoparticles. It also confirms that there is no contamination with the carbon NPs.

XRD Analysis of carbon NPs

The crystalline structure of the synthesized carbon NPs in terms of XRD pattern analysis was portrayed in Fig. 3.

As depicted in Fig. 3, for the synthesized carbon NPs the XRD pattern was observed with 2θ values. The diffraction angles such as 20.78° , 28.30° , 31.36° , and 68.23° , are indexed to the crystal planes (311), (420), (422), and (951) respectively (JCPDS 81-2220). The average size of the synthesized carbon NPs was calculated using the following Scherrer's

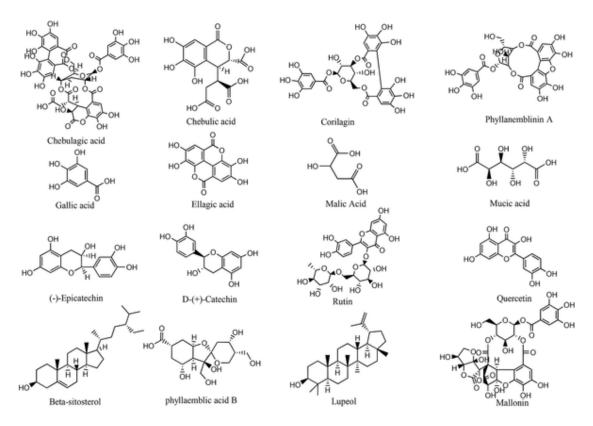


Fig. 1. Chemical components of Phyllanthus Emblica bark [13]

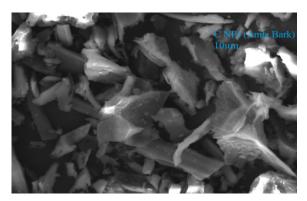


Fig. 2. SEM images of Carbon NPs

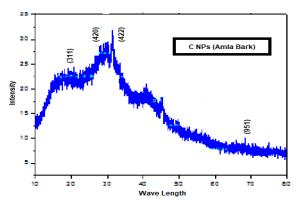


Fig. 3. XRD Pattern of Carbon NPs

Table 1. Physico-Chemical parameters of RW and BW without C NPs as adsorbent

Without C NPs	RW_1	RW ₂	RW ₃	BW ₁	BW ₂	BW ₃	BW ₄	BW ₅	BW ₆
Hardness	175	250	200	575	900	1350	600	1430	750
Chloride	55.08	62.96	70.83	566.63	338.40	424.97	369.88	582.37	354.14
Dissolved Oxygen	7.2	7.6	6.4	2.4	2.0	2.8	2.4	2.6	2.2
Alkalinity	80	70	160	340	200	240	280	250	230
TDS	45	67	178	195	934	152	555	124	232
pН	7.8	8.0	8.2	8.5	7.9	7.9	8.7	8.5	8.4
Ca	35.07	50.10	40.08	115.23	180.36	270.54	120.24	290.58	150.30
Mg	42.02	60.03	48.02	138.06	216.09	324.14	144.06	348.5	180.08

equation [14-16].

$$D = \frac{k\lambda}{\beta \cos\theta} \tag{1}$$

where,

D - Particle in size,

k – Scherrer's coefficient,

 λ – Wavelength of the X-ray source (1.5406nm),

 β – Full-Width Half Maximum (FWHM) and

 θ – diffraction angle

Based on Scherrer's equation, the average size of carbon NPs is estimated as 2.62nm.

During hard water treatment, the various hard

water analysis parametric values for Day 0 without C NPs are listed in Table 1.

The hard water treatment with C NPs on 5 consecutive days, and the measured parametric values for these days are mentioned in Tables 2-6 respectively.

As depicted in Fig. 4, the initial concentration of hardness for the collected water samples such as RW₁, RW₂, RW₃, BW₁, BW₂, BW₃, BW₄, BW₅, and BW₆ are 175, 250, 200, 575, 900, 1350, 600, 1450 and 750 respectively, after treated with C NPs (after 5 days) these values gradually decrease to 40, 35, 110, 350, 635, 1050, 355, 1175 and 545 respectively.

Table 2. Physico-Chemical parameters with C NPs as adsorbent (Day 1) $\,$

With CNPs (DAY 1)	RW_1	RW_2	RW ₃	BW_1	BW_2	BW_3	BW ₄	BW ₅	BW ₆
Hardness	100	125	175	475	750	1200	525	1365	690
Chloride	47.22	55.08	62.96	558.76	380.53	385.62	354.14	566.63	338.40
Dissolved	0.2	0.0	6.0	2.6	2.2	2.2	2.0	2.0	2.6
Oxygen	8.2	8.0	6.8	2.6	2.2	3.2	2.8	2.8	2.6
Alkalinity	50	50	60	160	190	240	270	240	210
TDS	37	65	177	195	931	152	523	105	221
pН	7.8	7.9	8.1	8.4	7.9	7.9	8.5	8.3	8.3
Ca	20.04	25.05	35.07	95.19	150.30	240.48	105.21	273.05	139.78
Mg	24.01	30.01	42.02	114.05	180.08	288.12	126.05	327.13	165.06

Table 3. Physico-Chemical parameters with C NPs as adsorbent (Day 2) $\,$

With									
C NPs	RW_1	RW_2	RW_3	BW_1	BW_2	BW_3	BW_4	BW_5	BW_6
(DAY 2)									
Hardness	100	75	150	425	725	1175	480	1310	655
Chloride	39.45	47.22	55.08	527.28	314.79	369.88	346.27	550.89	330.53
Dissolved	8.4	8.6	7.4	3.0	2.8	3.4	3.4	3.2	2.0
Oxygen	8.4	8.0	7.4	3.0	2.8	5.4	3.4	5.2	3.0
Alkalinity	20	50	60	150	190	230	250	220	200
TDS	29	64	170	194	926	152	496	88	198
pН	7.7	7.8	8.0	8.4	7.9	7.8	8.2	8.2	8.1
Ca	20.4	15.03	30.06	85.17	145.29	235.47	95.19	260.52	130.26
Mg	24.01	18.01	36.02	102.04	174.07	282.12	114.05	312.13	156.07

Table 4. Physico-Chemical parameters with C NPs as adsorbent (Day 3)

With									
CNPs	RW_1	RW_2	RW_3	\mathbf{BW}_1	\mathbf{BW}_2	\mathbf{BW}_3	$\mathbf{BW_4}$	\mathbf{BW}_{5}	BW_6
(DAY 3)									
Hardness	75	65	135	400	700	1125	425	1265	615
Chloride	31.5	39.5	39.4	519.41	306.93	362.01	322.67	527.28	314.7
Dissolved Oxygen	8.8	9.0	7.8	4.6	3.0	3.6	4.0	3.8	3.8
Alkalinity	20	25	50	150	190	230	230	210	200
TDS	29	62	159	193	913	152	471	75	163
pН	7.7	7.8	8.0	8.4	7.8	7.8	8.0	8.0	7.7
Ca	15.0	12.5	27.6	80.16	140.3	22.5.	85.17	255.5	122.7
Mg	18.0	15.0	33.0	96.04	168.07	270.11	102.04	306.12	147.06

Table 5. Physico-Chemical parameters with C NPs as adsorbent (Day 4) $\,$

With CNPs (DAY 4)	RW_1	RW ₂	RW ₃	BW_1	BW ₂	BW ₃	BW ₄	BW ₅	BW ₆
Hardness	50	50	125	375	650	1100	390	1205	590
Chloride	23.6	31.5	39.4	503.7	299.1	346.3	306.9	511.5	299.1
Dissolved Oxygen	9.8	9.6	8.0	4.8	3.6	4.0	4.8	4.8	4.6
Alkalinity	20	20	40	140	190	220	220	210	190
TDS	27	61	153	178	902	134	443	63	142
pН	7.5	7.8	7.9	8.3	7.8	7.8	7.7	7.8	7.7
Ca	10.0	10.0	25.1	75.15	130.3	220.4	77.66	240.5	117.74
Mg	9.00	9.00	27.0	84.04	150.1	252.1	84.04	282.1	132.1

With CNPs (DAY 5)	RW_1	RW ₂	RW ₃	BW_1	BW_2	BW ₃	BW ₄	BW ₅	BW ₆
Hardness	40	35	110	350	635	1050	355	1175	545
Chloride	15.74	15.74	31.48	495.80	283.32	338.40	291	503.67	283.32
Dissolved Oxygen	10.6	10.4	8.6	7.6	5.0	5.0	6.8	5.8	6.0
Alkalinity	15	20	40	130	160	180	200	190	170
TDS	23	61	147	153	899	131	407	55	129
pН	7.3	7.6	7.7	8.1	7.3	7.7	7.6	7.5	7.5
Ca	7.52	7.52	22.55	70.14	125.25	210.42	70.14	235.47	110.22

Table 6. Physico-Chemical parameters with C NPs as adsorbent (Day 5)

Hardness of Water Samples (Before & After Adsorption of C NPs)

150.06

252.11

84.04

282.12

132.05

84.04

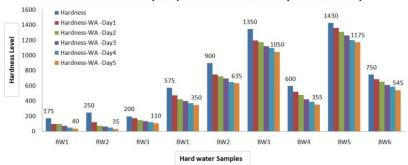


Fig. 4. Hardness of Water Samples

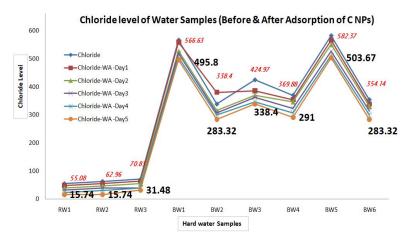


Fig. 5. Chloride level of Water Samples

As shown in Fig. 5, the initial amount of chloride present in the collected water samples are 55.08, 62.98, 70.83, 566.63, 338.40, 424.97, 369.88, 582.37 and 354.14 respectively, after being treated with C NPs (after 5 days) these values gradually decrease to 15.74, 15.74, 31.48, 495.80, 283.32, 338.40, 291.19, 503.67 and 283.32 respectively.

It was observed that (Fig. 6), at first the dissolved oxygen (DO) for the water samples are 7.2, 7.6, 6.4, 2.4, 2.0, 2.8, 2.4, 2.6, and 2.2 respectively, after

treatment the DO is significantly increased as 10.6, 10.4, 8.6, 7.6, 5.0, 5.0, 6.8, 5.8 and 6.0 respectively.

Before adding C NPs, the values of alkalinity for RW_1 , RW_2 , RW_3 , BW_1 , BW_2 , BW_3 , BW_4 , BW_5 , and BW_6 are 80, 70,160, 340, 200, 240, 280, 250, and 230 then adding C NPs the alkalinity reduced to 15, 20, 40, 130, 160, 180, 200, 190 and 70 respectively.

Before the treatment of water samples, the TDS (total dissolved solids) values are 45, 67, 178, 195, 934, 152, 555, 124, and 232. On the 5th day of



Mg

9.00

9.00

27.01

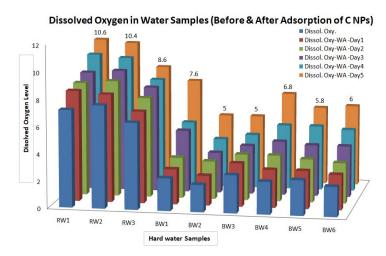


Fig. 6. Dissolved Oxygen level of Water Samples

Alkalinity of Water Samples (Before & After Adsorption of C NPs)

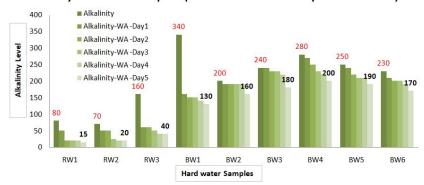


Fig. 7. Alkalinity of Water Samples

TDS of Water Samples (Before & After Adsorption of C NPs)

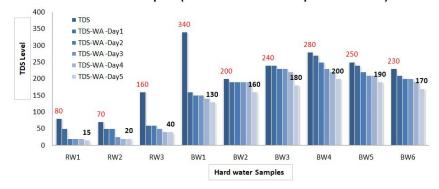


Fig. 8. TDS values of Water Samples

treatment, the TDS of water samples are 15, 20, 40, 153, 899, 131, 407, 55, and 129. From these values as explored in Fig. 8, TDS improvement in the hard water treatment has been confirmed.

Before treatment, the pH of water samples are 7.8, 8.0, 8.2, 8.5, 7.9, 7.9, 8.7, 8.5, and 8.4 falls to 7.3, 7.6, 7.7, 8.1, 7.3, 7.7, 7.6, 7.5 and 7.5 after 5^{th} day, as confirmed by Fig. 9.

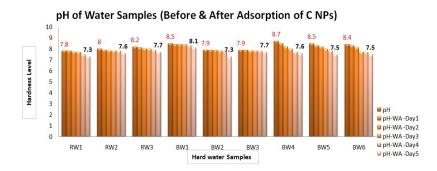


Fig. 9. pH values of Water Samples

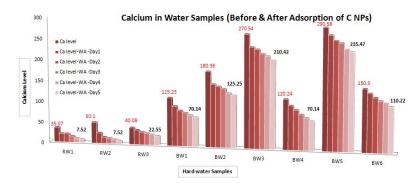


Fig. 10. Calcium concentration of Water Samples

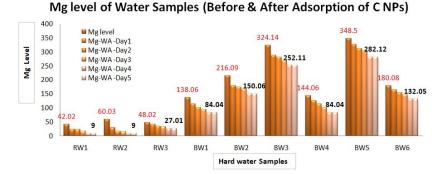


Fig. 11. Magnesium concentration of Water Samples

After 5 days the concentration of calcium in the water samples decreases from 35.07, 50.10, 40.08, 115.23, 180.36, 270.54, 120.24, 290.58, and 150.30 to 7.52, 7.52, 22.55, 70.14, 125.25, 210.42, 70.14, 235.47 and 110.22.

Magnesium concentration also decreases after treatment with C NPs as confirmed by values portrayed in Fig. 11. Without C NPs it was 42.02, 60.03, 48.02, 138.06, 216.09, 324.14, 144.06, 348.15 and 180.08. Whereas, with C NPs on the 5th day it decreased to 9.0, 9.0, 27.01, 84.04, 150.06, 252.11, 84.04, 282.12, and 132.05 respectively.

CONCLUSION

In the present study, an attempt was made to assess the physicochemical nature of rain-water samples and bore-well water samples before and after treatment with Phyllanthus Emblica. From the results of this study, it is understood that Phyllanthus Emblica wood reduces the TDS, hardness, calcium, magnesium, and chloride and increases the dissolved Oxygen levels. This analysis was carried out only for 5 days. But in the actual process, they simply cut the bark of the tree and put it in the well of the hard water. Here as the water



contains medium hardness the analysis was carried out for 5 days only. Suppose the water contains more hardness then the study may be extended to a greater number of days. In the future, Phyllanthus Emblica nanoparticles may be composite with Strychnos potatorum (Thethankottai) nanoparticles to produce clear and safe water.

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CONFLICTS OF INTEREST

The author declares that there are no conflicts of interest regarding the publication of this paper.

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