

Activated coconut coir for removal of water hardness

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ABSTRACT

Phosphoric acid activated coconut coir (ACC) has been used as an efficient and low cost filter material for water softening applications. The material has efficiently removed total hardness and calcium hardness from both artificial and natural hard water whereas the Mg hardness removal efficiency was relatively lower than the other two hardness values. The optimum conditions for maximum hardness removal were; ACC dose 80.00 g L⁻¹, stirring time 30 min and settling time 2 h. Under the optimized conditions, the maximum removal efficiencies of total, Ca and Mg hardness, were approximately 46%, 66% and 30%, respectively from artificial hard water. More efficient softening was observed when the hard water was boiled to remove the temporary hardness prior to filtering through the ACC medium. It was also, observed that the hardness removal efficiency was higher when two layers of ACC together with a sand layer were used. The results indicate that ACC has potential in future water softening applications.

Keywords: Activated coconut coir; Hardness; Water softening; Temporary hardness; Permanent hardness

1. Introduction

Water plays a major role in absorbing minerals, particularly, calcium and magnesium into the human body. Inadequate supply of either ion can cause health problems. However, excessive intake of both ions is also harmful; excessive intake of calcium causes kidney stones and that of magnesium leads to diarrhea and laxative effects [1]. In addition, hard water causes many difficulties at domestic and industrial levels leading to scaling in boilers, washing machines and pipes, difficult lathering with soap, undesirable spots on sinks and clothes as well as toughening of skin and hair.

The principal natural sources of hardness in water are dissolved polyvalent metallic ions leaching from sedimentary

rocks, seepage and runoff from soils. Calcium and magnesium are the most common ions found in sedimentary rocks such as limestone. These limestone deposits are mainly responsible for water hardness [2,3].

Various techniques ranging from conventional methods to modern technologies are being used in water softening applications. Among them, ion-exchange [4,5], membrane filtration [6], electrochemical techniques [6–8], and adsorption by natural adsorbents [9–13] are commonly practiced. In ion exchange method, calcium and magnesium ions in water are exchanged with sodium ions in the resins. However, the major drawback in such resins is that sodium ions released to the medium subsequently, causes serious health problems. On the other hand, although the ion-exchange membrane techniques are very efficient they involve high energy consumption [6].

Electro-chemical techniques such as electrodialysis, electrodeionization (EDI), electro-deionization reversal (EDIR), electrocoagulation and capacitive deionization have been

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widely used in softening hard water. Su and co-workers have developed an advanced EDI process with improvements in resin packing and reduced 99.8% water hardness by using six EDI stacks with different inner configurations [7]. On the other hand, in EDIR technique, average removal efficiencies of cations of more than 88% have been achieved [7]. Furthermore, seawater osmotic dilution method has been investigated recently as an efficient method for water softening [9]. These techniques used in water softening applications are not feasible for developing countries due to the high energy costs involved in operation [10].

Plant materials have also been used in few studies as low cost options in water softening applications. The capillary effect of kenaf fibers (*Hibiscus cannabinus* L.) has been used to remove 53% of hardness from very hard water [10]. Some of the natural adsorbent materials that have so far been tested in hardness removal are pumice stone [11], *Moringa oleifera* seed [12] and peanut hull [13]. Sepehr et al. found that alkali modified and unmodified pumice stones are effective low cost adsorbents which demonstrated high efficiency in hardness removal applications where the total hardness was reduced to 300 mg L⁻¹ in natural hard water [11].

Furthermore, *Moringa oleifera* seeds have been used to remove both Ca²⁺ and Mg²⁺ from natural water [12], however, the maximum adsorption of these ions was observed at acidic pHs imposing certain practical limitations due to the basicity of natural hard water.

Although, most of the techniques developed so far show significant efficiency in removal of hardness in water, high energy consumption is a major drawback, making them unsustainable approaches for many countries. As a result, there is an increased scientific interest to develop more eco-friendly, sustainable and economical methods for water hardness removal and this aspect of research has received low scientific attention thus far.

In this regard, activated carbon which has superior adsorption capacity for many water pollutants can be regarded as a promising alternative. We have earlier reported the ability of activated coconut coir in removing sodium, magnesium and fluoride ions from both natural and artificial water [14–16]. This study further attempts to investigate the potential of activated coconut coir prepared by the same method, in water softening applications, thus qualifying the materials as a cheap, sustainable and a multi-purpose adsorbent. The efficacy of activated coconut coir in removing both temporary and permanent hardness is investigated.

2. Materials and Methods

2.1. Materials

All chemicals used were of analytical grade and purchased from BDH Chemicals Ltd. All solvents were distilled prior to use. Metal ion salts of CaCl₂ (99 wt% assay), MgSO₄·7H₂O (99 wt% assay) and NaHCO₃ (98 wt% assay) were used without further purifications. Artificial hard water, pH 10 buffer, HHSNNA indicator (3-Hydroxy-4-(2-hydroxy-4-sulfo-1-naphthylazo)-2-naphthalenecarboxylic acid)/calcium indicator (solid), hardness indicator (solid), 0.01 M standard EDTA (ethylene di-amine tetraacetic acid) were used in the analysis of hardness in water.

Raw coconut coir was collected from Rambukkana, Sri Lanka. Natural hard ground water samples were collected from dug wells in Madampe, Mathale, Anuradhapura, and Kirimatiyawa areas in Sri Lanka.

2.2. Methods

2.2.1. Preparation of artificial hard water

Artificial hard water was prepared by mixing 288.0 mg of CaCl₂, 220.0 mg of MgSO₄·7H₂O and 390.0 mg of NaHCO₃ in 1 dm³ of distilled water and stored in a plastic bottle [17–19].

2.2.2. Determination of water hardness

The physical parameters, such as, pH, conductivity, total dissolved solid content, salinity of hard water samples were determined. Water hardness was determined using ASTM D-1126 [20] standard method.

2.2.2.1. Determination of total hardness

Test water sample (25.00 cm³) was diluted to 50.00 cm³ with distilled water, added 4.00 cm³ of pH 10 buffer and 0.20 g of Erichrome Black T indicator and titrated against 0.01 mol dm⁻³ EDTA solution until the color changed from purplish red to blue [20].

2.2.2.2. Determination of calcium hardness

Test water sample (25.00 cm³) was diluted up to 50.00 cm³ with distilled water, added 2.00 cm³ of NaOH, stirred and kept for 3 min. Then, HHSNNA indicator (0.200 g) was added and titrated against 0.01 mol dm⁻³ EDTA solution until the color changed from red to royal blue [20].

All three types of hardness values were calculated as CaCO₃ in ppm according to the ASTM standards (D 1126-02) [20].

$$\text{Total Hardness as CaCO}_3 = \frac{(1000)(c)}{S} \quad (1)$$

where c = standard EDTA titre in mL (for total hardness determination); S = sample size in mL.

$$\text{Calcium Hardness as CaCO}_3 = \frac{(1000)(D)}{S} \quad (2)$$

where D = standard EDTA titre in mL (for calcium hardness determination); S = sample size in mL.

$$\text{Magnesium Hardness as CaCO}_3 = \text{Total Hardness} - \text{Calcium Hardness} \quad (3)$$

2.2.3. Preparation of activated coconut coir (ACC)

The preparation and complete characterization of activated coconut coir were reported earlier by Hettiarachchi et al. [14]. Dried coir dust (30.00 g) was soaked in H₃PO₄ acid, (1.00 mol dm⁻³, 50 wt%), and boiled for 1 h. The solution was allowed to stay in an oven at 100°C for 48 h. The sample was then, pyrolyzed at 450°C for 1 h, cooled down

to room temperature and washed repeatedly with distilled water until the pH 7 was reached in the supernatant. It was oven dried at 100°C for 48 h before use.

2.2.4. Hardness removal by ACC

2.2.4.1. Mass optimization

Artificial hard water sample (200 cm³) and 4.00 g of powdered ACC were equilibrated by stirring at 500 rpm for 20 min. Then, it was allowed to settle for 3 h and the solution was filtered through a sintered crucible. The procedure was repeated with 200 cm³ of artificial hard water samples treated with 4.00, 8.00, 12.00, 16.00, 20.00 g of ACC under the same stirring and settling times. The filtrates were analyzed for total, calcium and magnesium hardness using EDTA method as explained above.

2.2.4.2. Time optimization-stirring time and settling time

Artificial hard water sample (200 cm³) and 16.00 g of powdered ACC were equilibrated by stirring at 500 rpm for 10 min and allowed to settle for 3 h. The procedure was repeated with 5 artificial hard water samples while stirring for different time intervals, 20 min, 30 min, 40 min, 50 min, 60 min. The filtrates were analyzed for total, magnesium and calcium hardness using EDTA method.

The stirring time to get the highest removal of hardness was 30 min.

The above procedure was repeated with 5 artificial hard water samples at different settling times of 2 h, 3 h, 4 h, 5 h and overnight. The filtrates were analyzed for total, magnesium and calcium hardness using EDTA method.

The settling time to obtain the highest removal of hardness was 2 h.

After optimizing the experimental conditions, such as, ACC dosage, stirring and settling times for maximum hardness removal, rest of the experiments were carried out under optimized conditions, 16.00 g of ACC in 200 cm³ of water, 30 min stirring time and 2 h settling time. For comparison, the above test was repeated using commercial activated carbon derived from coconut shell.

2.2.5. Permanent hardness removal from the artificial hard water

2.2.5.1. Determination of temporary and permanent hardness of artificial hard water

In order to remove the temporary hardness, artificial hard water (900 cm³) sample (label as S_{hard}) was boiled for an hour and cooled to room temperature. Boiling and cooling were repeated for another 2 times and the sample was filtered through a sintered crucible (label as S_{boiled}). Then, the hardness of the filtrate was determined using EDTA method.

2.2.5.2. Determination of permanent hardness removal efficiency of synthesized ACC

Hard water sample S_{boiled} (200 cm³) after removing the temporary hardness, was stirred with 16.00 g of powdered ACC for 30 min and allowed to settle for 2 h. Then, the sample was filtered through a sintered crucible (label as $S_{\text{filt-ACC}}$) and the hardness of the filtrate was determined using EDTA method.

For comparison, the above procedure was repeated using hard water sample S_{boiled} using coconut shell based commercial activated carbon and the hardness of the filtrate (label as $S_{\text{filt-AC}}$) was determined using EDTA method.

2.2.5.3. Total hardness removal by synthesized ACC after repeated filtration using a filter bed

A filter bed was constructed by packing clean sand layer (washed thoroughly and dried) in between two layers of ACC (80.00 g each). Then, artificial hard water (600 cm³) was passed through the filter bed for 3 h. The filtrate was collected and titrated against EDTA to determine the hardness. The procedure was repeated with only a sand filter bed in order to confirm that sand has no contribution to hardness removal process.

2.2.6. Total hardness removal from natural hard water using ACC

Initial hardness of the natural hard water collected from Madampe, Sri Lanka, was determined. Then, a natural hard water sample (200 cm³) was stirred with 16.00 g of powdered ACC for 30 min and allowed to settle for 2 h. The sample was filtered through a sintered crucible and the hardness of the filtrate was determined using EDTA method. The same procedure was followed for the other natural hard water samples collected from Mathale, Anuradhapura and Kirimatiyawa.

In all experiments the stirring speed was kept constant at 500 rpm and the temperature was 25°C. All experiments were triplicated.

3. Results and Discussion

Physical and chemical characteristics of the ACC prepared by phosphate activation method, have paramount importance in deciding its suitability for water softening applications. The detailed characterization of the ACC is reported by Hettiarachchi et al. in a previous publication [14]. The ability for adsorption of cations such as Ca²⁺ and Mg²⁺ is closely related to the pore size, point of zero charge (PZC) and functional groups available in the pores of ACC. It was found that the PZC for ACC is 5.5 ± 0.1 making it suitable for adsorption of cations from basic solutions. On the other hand, high BET surface area (1235 m² g⁻¹) and the presence of nano-size pores further qualify the material for water softening applications. In addition, the presence of functional groups such as hydroxyl and phosphate facilitate the adsorption of cations into the pores. It is noteworthy that although the same activation process as for coconut shell is used in this study the temperature history, PZC and the surface functionality are different for ACC, thus making it suitable as a low cost filter material for removal of water hardness.

3.1. Conditions optimization

The optimization of contact time and the amount of adsorbant (ACC) on hardness removal were carried out using artificial hard water.

3.1.1. Mass

The hardness removal efficiency of ACC increased with the increase of ACC dose and the maximum percentage removal was observed at 16.00 g of ACC per 200 cm³ hard water (Fig. 1).

3.1.2. Time

The optimum time for stirring and settling to reach the maximum adsorption equilibrium have been studied.

3.1.2.1. Stirring time

Fig. 2 shows the variation of percentage removal of hardness as a function of stirring time. The maximum hardness removal was obtained at 30 min of stirring time. However, when stirring was continued for more than 40 min, percentage hardness removal decreased drastically. Furthermore, Mg adsorption efficacy was shown to be significantly lower than that of calcium.

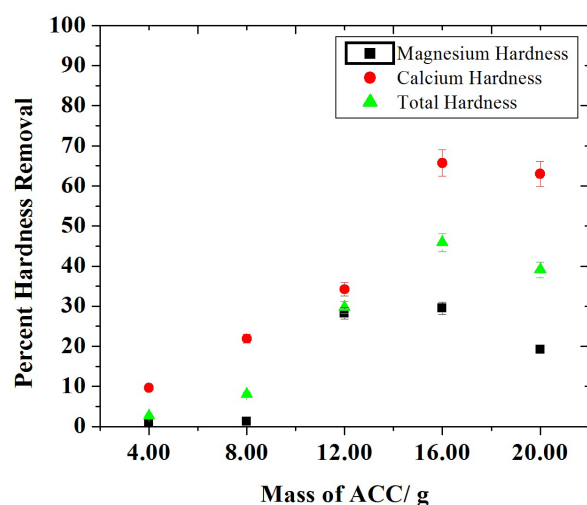


Fig. 1. Mass optimization of ACC for artificial hard water.

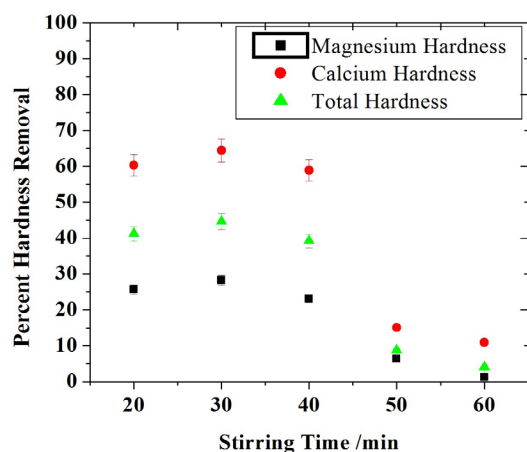


Fig. 2. Stirring time optimization.

It has been previously reported by Hettiarachchi et al. that the cation adsorption on ACC proceeds through a physisorption process which obeys Freundlich isotherm [14]. The initial increase in the adsorption capacity may be due to the availability of large number of negatively charged pores initially and, the subsequent decrease may be explained by referring to the desorption that occurs as a result of mechanical forces applied. Furthermore, after a certain extent of surface coverage, electrostatic hindrance exerted by the surface adsorbed cations attempt to release some amount of cations from the surface. The process may either be diffusion controlled desorption or dissolution.

3.1.2.2. Settling time

Fig. 3 illustrates the variation of percentage removal of hardness with settling time. The best settling time for the maximum removal of hardness was found to be 2–3 h. However, when the settling time was increased above this duration the percentage hardness removal was decreased gradually till 5 h of settling time. When the hard water was kept in contact with the adsorbant for overnight the percentage hardness removal was further decreased. This observation can be explained referring to the desorption phenomena which occurs after attaining the equilibrium conditions. The fact that ACC takes a shorter time to achieve adsorption equilibrium is another advantage of ACC to be used as a filter material in domestic filters.

3.2. Analysis of artificial hard water under optimized conditions

The efficacy of ACC in softening artificial hard water was studied under the optimized conditions; 16.00 g ACC, 30 min stirring time and 2 h settling time, and 200 cm³ of hard water.

The variations of some physical parameters and three types of hardness of artificial hard water before and after treatment with ACC were determined and tabulated in Table 1.

Table 1 compares the values of pH, salinity, conductivity, TDS, and three types of hardness of laboratory prepared artificial hard water before and after treatment with ACC.

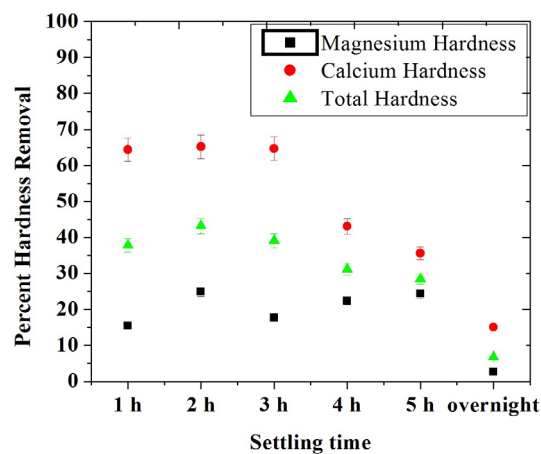


Fig. 3. Settling time optimization.

Table 1
Physical parameters of artificial hard water before and after treatment with ACC

Parameter	Artificial hard water before treatment	Artificial hard water after treatment with ACC	Distilled water
pH	10.1	8.6	6.8
Salinity	540 ppm	1 ppt	2 ppm
Conductivity (μS)	1072	2110	3.2
TDS	711.0 ppm	1.4 ppt	2.1 ppm
Total hardness as CaCO_3/ppm	296	160	Not detected
Calcium hardness as CaCO_3/ppm	146	50	Not detected
Magnesium hardness as CaCO_3/ppm	156	110	Not detected

Artificial hard water used for the study is highly basic. After treatment through the filter material pH and other parameters, such as, salinity, TDS and hardness have been significantly decreased. The value of TDS decreased after the treatment with ACC, because other ions like sodium, present in the water was also adsorbed onto ACC. As a result, the amount of total dissolved solids in the sample is decreased. This is an added advantage of ACC over other natural filter materials in water softening applications as such some natural adsorbents excrete minerals, metal ions, etc. to the filtrate increasing TDS. However, removal of Mg hardness is relatively lower than that of Ca hardness. As sodium ions are also present with the calcium and magnesium ions, magnesium ions have to compete with other two types of ions during the adsorption process. This observation can be explained referring to the ionic radii and the size of the solvation sphere. Sodium and calcium ionic radii are almost similar (116 ppm for sodium and 118 ppm for calcium) while that of magnesium ion is 86 ppm. Smaller cation size of Mg leads to higher solvation capacity thus, bulky Mg ion prefers to stay in the solution rather than adsorbing on to ACC. Similar observation was reported by Hettiarachchi et. al. for desalination of sea water using the same ACC.

Under optimized conditions, as shown in Table 1, removal efficiency of total hardness, calcium hardness and magnesium hardness are 46%, 66% and 30%, respectively, where, very hard water was converted to moderately hard water. In the presence of competitive ions, the adsorption efficiency of Mg is low. The observations agree well with our previous reports where Mg ion adsorption was minimum when Na ions were present.

In order to highlight the novelty and the unique water hardness removal characteristics of ACC, the experiment was repeated with coconut shell activated carbon, (Fig 4). According to the Fig 4, it is evident that the coconut shell AC does not have the capacity for removal of Ca^{2+} and Mg^{2+}

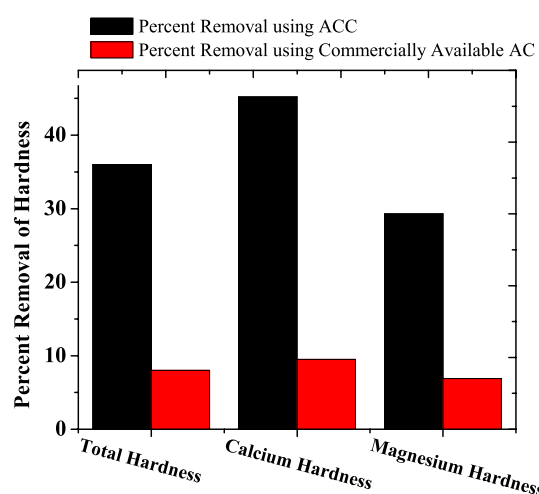


Fig. 4. Comparison of water hardness removal efficiencies of ACC and commercialcoconut shell AC.

from hard water. This observation is directly related to the structural features and the thermal history of ACC.

3.3. Determination of permanent hardness of the artificial hard water

Hardness in water is two-fold, permanent and temporary. The efficiency of ACC in removing permanent hardness of artificial hard water was determined after removing the temporary hardness after boiling. As given in the Table 2, the total hardness was removed by 38% after boiling while that of calcium and magnesium was removed by 53% and 17%, respectively. These observations confirm that the major contributor to permanent hardness is magnesium.

The artificial hard water, S_{hard} , is considered as very hard water, according to the WHO recommendations. As shown in Table 2, 38% of total hardness is removed by boiling. When compare the hardness of S_{boiled} and $S_{\text{Filt-ACC}}$, 36%

Table 2
Physical parameters of water samples

Parameter	S_{hard}	S_{boiled}	$S_{\text{filt-ACC}}$	$S_{\text{Filt-AC}}$
pH	9.2	9.6	8.9	9.6
Salinity	600 ppm	966 ppm	1 ppt	1 ppt
Conductivity	1185 μS	1932 μS	2 mS	3 mS
TDS	784 ppm	1275 ppm	1.26 ppt	1.71 ppt
Total hardness as CaCO_3/ppm	320	200	128	184
Calcium hardness as CaCO_3/ppm	180	84	46	76
Magnesium hardness as CaCO_3/ppm	140	116	82	108

of the total permanent hardness is removed using synthesized ACC. It effectively removes 45% of calcium and 29% of magnesium hardness from the permanent hardness.

Furthermore, when comparing the hardness in the original very hard water sample with that of the water sample after undergoing two-step filtering process ($S_{\text{filt-ACC}}$), it could be concluded that hardness of water was effectively removed by boiling and then filtering through synthesized ACC. Total hardness removal efficiency reached 60% while calcium hardness removal efficiency achieved 75%. Magnesium hardness removal efficiency was also increased up to 41% by this method suggesting the potential of using ACC in future household filters for water softening applications. For convenience of comparison, total hardness, calcium and magnesium hardness are plotted together for all 4 above samples in Fig. 5.

3.4. Comparison of the hardness removal efficiency of ACC: Two step method (boiling + filtering through ACC) vs filtering through ACC

Table 3 summarizes percentage removal of all three types of hardness by single filtration through ACC and in

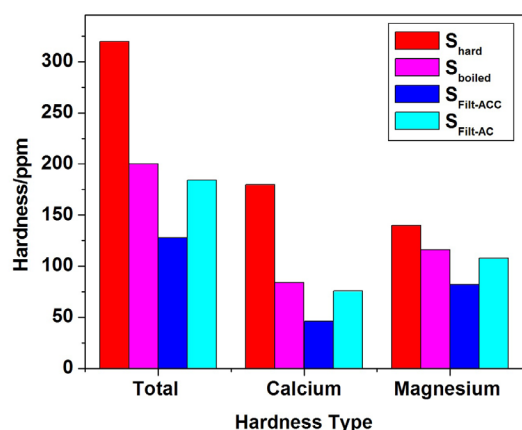


Fig. 5. Comparison of total, calcium and magnesium hardness in four samples.

Table 3
Comparison of the percent hardness removal efficiency of ACC in two step method and normal filter method

Parameter	Percentage removal by single filtration through ACC (1 step)	Percentage removal by boiling + single filtration through ACC (2 step)
Total Hardness as CaCO_3/ppm	46	60
Calcium hardness as CaCO_3/ppm	66	74
Magnesium hardness as CaCO_3/ppm	30	41

two-step process of boiling followed by single filtration through ACC. There is a significant reduction in the total hardness in the two-step method. Calcium and magnesium hardness are also considerably reduced in the two-step method.

3.5. Determination of total hardness removal efficiency of the water filter containing two ACC layers and a sand layer

The efficiency of removing hardness from artificial hard water using a water filter constructed using one sand layer and two beds of ACC (80.00 g) was determined.

The physical parameters and three types of hardness of the artificial water and the filtrate after passing through the constructed filter are shown in Table 4. The filter has 51 % efficiency of removing the total hardness after passing through a sand layer and two ACC layers. The calcium and magnesium hardness have also been considerably removed, 56% and 48%, respectively. In the two-step process, water hardness was reduced down to moderate level from very hard level. If the hard water used was without temporary hardness, the removal efficiency increases further.

3.6. Determination of total hardness removal efficiency of natural hard water using ACC

The synthesized ACC was tested for its capability in reducing the hardness in natural hard water samples collected from 4 locations in Sri Lanka. The samples were collected from dug wells in 4 different areas of the island of which hardness was found to be varying from hard to very hard level due to variety of geo-morphological reasons. The quality of the collected water samples is shown in Table 5. All water samples are highly alkaline with high salinity, conductivity and TDS.

All water samples demonstrated very high level of hardness prior to treatment. However, single filtration through

Table 4
Physical properties of the artificial hard water and filtrate collected after passing through the constructed filter bed

Parameter	Artificial hard water	Filtrate	Percent removal of hardness
pH	10.1	8.9	–
Salinity	540 ppm	1 ppt	–
Conductivity	1072 μS	2 mS	–
TDS	711 ppm	2 ppt	–
Total hardness as CaCO_3/ppm	296	144	51
Calcium hardness as CaCO_3/ppm	146	64	56
Magnesium hardness as CaCO_3/ppm	156	80	48

Table 5
Physical parameters of natural hard water

Parameter	Madampe	Mathale	Anuradhapura	Kirimatiyawa
pH	>11.0	>11.0	>11.0	10.2
Salinity/ppm	740	621	782	527
Conductivity	Very high	Very high	Very high	1089 μ S
TDS/ppm	1254	843	1301	708
Total hardness as CaCO ₃ /ppm	798	622	810	312
Calcium hardness as CaCO ₃ /ppm	432	340	439	150
Magnesium hardness as CaCO ₃ /ppm	366	282	371	162

the ACC removed the total hardness by ~30%. The results are summarized in Table 6. If the used ACC is regenerated by the method suggested by Hettiarachchi et al. [14], ACC can be repeatedly used for water softening at a low cost at domestic level.

The results suggest that the ACC has high promise as a low cost filter material which has the capacity for use in water softening applications in addition to the desalination and deflouridation applications reported before. Thus, unlike other low cost materials reported for water purification applications thus far, ACC prepared by phosphoric acid activation method has the future potential as a multi-purpose filter material.

4. Conclusions

ACC prepared by phosphoric acid activation method demonstrates the potential for water softening applications.

The optimum parameters for the maximum adsorption efficiency has been established. After establishing the suitable conditions, the experiments are extended to natural hard water samples. ACC shows higher affinity towards the removal of Ca²⁺ compared to that of Mg²⁺. Hardness removal efficiency was relatively higher for artificial hard water compared to that of natural hard water. However, during a single filtration naturally very hard water was converted to a moderately hard water.

The percentage removal of calcium, magnesium and total hardness by ACC is enhanced when the water is boiled to remove the temporary hardness prior to the filtering process. Furthermore, the filter is more efficient when two layers of ACC and a layer of sand was used to carry out repeated filtration. Therefore, it is concluded that activated coconut coir based filters have potential in future water softening applications. A domestic water filter containing a filtering system with two or more layers of ACC coupled with a prior boiling step is a cheap, durable and eco-friendly system for

Table 6
Hardness removal efficiency of ACC from natural hard water

Parameter	Initial value	Final value	% removal
<i>Madampe</i>			
Total Hardness as CaCO ₃ /ppm	798	546	32
Calcium hardness as CaCO ₃ /ppm	432	274	37
Magnesium hardness as CaCO ₃ /ppm	366	272	26
<i>Mathale</i>			
Total Hardness as CaCO ₃ /ppm	622	412	34
Calcium hardness as CaCO ₃ /ppm	340	250	27
Magnesium hardness as CaCO ₃ /ppm	282	162	43
<i>Anuradhapura</i>			
Total Hardness as CaCO ₃ /ppm	810	552	32
Calcium hardness as CaCO ₃ /ppm	439	292	33
Magnesium hardness as CaCO ₃ /ppm	371	260	30
<i>Kirimatiyawa</i>			
Total Hardness as CaCO ₃ /ppm	312	210	33
Calcium hardness as CaCO ₃ /ppm	150	103	31
Magnesium hardness as CaCO ₃ /ppm	162	107	34

water softening applications. More importantly, when these findings are coupled with the previous work on the potential of the same ACC in desalination and defluoridation applications, a multi-ion adsorption filter can be developed.

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