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Assessing the effectiveness of low-cost materials to act as filter media for phosphate sorption

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ABSTRACT

In this research, an assessment was conducted for several materials to examine their efficiency for sorption phosphate and their potential use as filter media. This work focuses on seeking materials that can achieve high, rapid, and robust phosphate retention. To assess the phosphate removal efficiency, batch-mode testing was conducted. The capability of the selected materials for effectively removal the phosphate is largely influenced by their inherent properties. Therefore, this research determines the effectiveness of the material by examining its physical and chemical characteristics. The selection method for these materials was guided by the understanding that the sorbents that have the most effective behavior for phosphate tend to contain easily soluble Ca/Mg compounds or Fe/Al hydroxides. The findings of the batch experiments demonstrated that material such as Bio ash (Bio-A) exhibited superior phosphate removal performance throughout the entire duration of the experiments. In contrast, materials calcium carbonate (C-C) and conventional silica (C-S) displayed relatively poor removal performance compared to Bio-A.

1. Introduction

Urban and industrial societies produce significant quantities of wastewater, which poses an ongoing threat to the quality of freshwater globally [1]. The global standards for environmental protection have become more stringent, with distinguished regulations, according to Ntifo [2] the regulations were announced in 2000 by the European Water Framework Directive. Consequently, it is imperative to avoid direct discharge of wastewater into water bodies and implement impurity removal processes to mitigate the adverse impacts on the ecosystem and human public health. Krishnaswamy et al. [3] identified the phosphate as one of the main elements of the nutrients. Phosphorus is discharged into aquatic ecosystems due to several human activities.

These activities can be broadly categorized into two categories: point sources, such as wastewater treatment plants, and diffuse sources, like agricultural runoff [4]. Typically, approximately 1-2 mg/l *of* phosphorus possible to be eliminated by the secondary treatment [5], resulting in excessive phosphorus release in the ultimate effluent. To address this issue, various tertiary treatment technologies have been introduced, including adsorption of activated carbon, precipitation technique and oxidation of chemicals, constructed wetlands, and membrane filtration with the assistance of coagulants [6]**.** To reduce the negative effects of phosphorus loading, numerous wastewater treatment plants have adapted or upgraded their schemes to include tertiary treatment unit to achieve removing the phosphate.

Neethling et al. [7] have declared the utilization of advanced physical, chemical, and biological methods will assist in achieving low levels of phosphate concentration at 0.2 to 0.3 mg/l, with some processes demonstrating even superior performance. Acknowledging the imperative of treat a solution polluted with phosphate, the use of active material as filter media for phosphate capture has gained a significant attention in last decades as an appealing approach for treatment the effluents. Currently, there is a necessity to identify materials have strong affinity for nutrients such as phosphate to accomplish the desired quality effluents as mandated by the European Water Framework Directive WFD [8]. In light of these outcomes, many researchers have recognized the importance of exploring alternative materials as an environmental friendly strategies for capturing the phosphate. Based on Johansson West Holm [9], the sustainable materials can be categorized as: materials from natural resources, industrial or by-products materials, and manufactured materials. In recent decades, a considerable investigations have been conducted to assess the efficacy of Phosphate sorbing materials (PSMs) in removing phosphate from wastewater. Earlier studies have primarily focused on evaluating the removal capacity of these materials for elevated phosphate concentrations [10]. Phosphate can bind to Fe and Al oxides through surface complexation, including covalent bonding, resulting in the formation of robust complexes that are not easily adsorbed [11]. The process of adsorption includes sorption reactions occurring at the particle surface. This adsorption is specific to certain sites: an exchange occur between a several groups of hydroxyl from the surface of metal oxide with atoms from ion of phosphate, leading to formation of a bidentate/binuclear or monodentate / mononuclear. While the reaction is theoretically reversible, practically the absorbed phosphate is release at low level [12]. Phosphate molecules adsorb onto the $CaCO_3$ surfaces, and subsequently a secondary Ca-phosphate released as a precipitate [13] and [14]. Precipitation of Ca-phosphates occurs in phosphate solutions when the solubility product of the mineral is surpassed. At the moment that the product solubility of the mineral is exceed, a precipitation of Ca-phosphates occur in phosphate solutions. In situations where there are high concentrations of phosphate in the solution, such as when using a high phosphate concentration, a significant amount of Ca-phosphate can precipitate. A significant amount of Ca will dissolve from $CaCO_3$, as a result of precipitation process the Ca concentration will be decreased in the effluent [15] and [16]. As long as the concentrations of phosphate and soluble Ca exceed the solubility product of the precipitate, the process keeps continue. Selecting an appropriate filter media is a vital issue in the technology of filtration process to achieve the desired effluent quality [17]. Forces which sorted as chemical and physical is an essential elements for the reactivity between the particles of the media and the suspended impurities, they are the main factors in achieving a clean suspension. The forces involved in the filtration process, such as media particle size, effluent velocity, and suspended particle size, play a vital role in determining the filtration outcomes [18] and[19]. Numerous parameters are employed to investigate the hydraulic behavior of the effluent within filters. Among these parameters, particle size distribution stands out as a significant indicator of system efficiency, reliability, and durability. The aim of the research has been accomplished based on two central phases. Firstly, seeking for the eligibility of several materials to perform as phosphate filter media by subjecting these selected materials for chemical and physical experiments. Secondly, utilize the batch test to determine the most efficient material for phosphate removal.

2. Material and method

2.1 Prepartion and setyp

Various factors must be taken into consideration throughout the material selection method. According to [20], new research works should aim to establish an alternative method for determining the most effective and efficient media. The fundamental aspect lies in the characteristics of the material, as referred to previously in the introduction section, where materials containing Fe/Al and/or soluble Mg and Ca are expected to exhibit phosphate retention capabilities. The physical characteristics of filter material, including (shape, size, and porosity) have an outstanding impact on the surface reactivity of the materials and the hydraulic conductivity as well. A current defy for the managers in the industrial sector and the researchers in the filtration process field is to identify materials that align with sustainable practices. Consequently, the cost and availability of materials directly impact on the selection process. This research specifically concentrated on materials considered low-cost, such as natural materials or by-product waste materials, to meet the requirements of the environment and economy. The selected materials in this work whose capability for phosphate sorption was tested are Bio-Ash (Bio-A), calcium carbonate (C-C) and conventional silica (C-S). According to earlier research, C-C is considered a type of rock which Ca is dominated on its composition. The C-C contains a low amounts of Al, Fe, Mg and other oxides. Furthermore, the ash composition consist of small percentage of Mg and Ca, and the dominated compounds in ashes are Al and Fe. The C-S has been utilized as a filter media for phosphate removal for a long time. In comparison with other materials The C-S has a limited capability for phosphate removal. However, it is included in this work as one of the typical materials that utilize for comparison with outcomes.

2.2 Materials characteristics

The chemical and physical characteristics are essential to define the mechanism that allow material to remove the phosphate. Consequently, a characteristics' examination for the filter media serves as the initial stage in assessing the selected materials' adequacy for the filtration system. The description of filter material should exhibit rapid interaction and a propensity for phosphate sorption, along with a high capacity for phosphate retention. The outcomes of the analysis of material properties will aid in elucidating the factors contributing to the strong affinity between the filter material and phosphate ions. According to the study goal the below experiments will be the main identification tool for the materials' characteristics as below:

- Hydraulic conductivity K
- Specific gravity S.G
- Bulk density
- Porosity ε
- Particle size distribution PSD

Chemical characteristics

- X-ray fluorescence analyser XRF

2.3 Batch test procedure

Batch experiments were conducted to initially evaluate the phosphate sorption capacity of the chosen materials. A series of beakers (capacity: one-liter) were utilized to bring phosphate solution samples (at specific volumes) into contact with the same masses of the tested materials. In order to disallow the materials from floating upward when immersed in the solution, a liquid-to-solid ratio of 1.7 ml per gram was maintained. The suspension was initially shaken and then allowed to stand undisturbed throughout the period of the test to reduce the mixing impact on the outcomes of the sorption. Collection of samples were conducted at regular intervals, the time of collection gradually increasing as the experimental period progressed. To ensure more accurate outcomes, the collected samples were subjected to filtration process through a filter paper (size: 0.45 µm) to remove any suspended particles that could interfere with light

scattering when subjected to spectrophotometric analysis.

3. Results and discussion

3.1 Physical characteristics
3.1.1 PSD analysis
The composition of materials plays an essential role in determining their suitability as filter media. An analysis of PSD was conducted to assess the compatibility of the selected materials' particle size with the filtration systems. For four constituent the soil is categorized, one of these categorizes is the sand, defined as soil particles greater than 0.06 millimetres. Thus, the materials selected for this investigation are classified as sand. Sieve analysis procedure was employed to determine the distribution of the particle size for the collected samples, PSD is an appropriate approach if the sizes of the tested particle are larger than 0.075 mm, which is the case for our sand samples. The outcomes obtained through sieve analysis typically represented by proportions, the percentage of the samples' total weight which is passed through the sieves. A series of sieves, numbered 4, 6, 10, 16, 30, 50, and pan, were arranged in a top-down sequence. Subsequently, the way that samples were applied to the sieves by placed them from the top and then subjected to vibrations from a vibrator for a period of 10 minutes.

Table 1. Sieve analysis outcomes for C-S.

Sieve No.	4	6	10	16	30	50	Pan
Diameter mm	4.7	3.3	2	1.1	0.6	0.3	14
Retained mass g	0	0	56	168	315	147	2
Retained %	0	0	8	24	45	21	100
Cumu. retained %	0	0	8	32	77	98	0
Finer %	100	100	92	68	23	2	14

Table 2. Sieve analysis outcomes for C-C.

Sieve No.	4	6	10	16	30	50	Pan
Diameter mm	4.7	3.3	2	1.18	0.6	0.3	
Retained mass g	0	0	144.8	393.4	140.6	1.1	20
Retained %	0	0	20.6	56.2	20	0.16	2.86
Cumu. retained %	0	0	20.6	76.8	96.9	97.1	100
Finer %	100	100	79.3	23.1	3	2.8	0

Table 1 presents the findings of the sieve analysis for the sample of C-S, indicating the majority of the samples' mass was retained in sieve number 30, and accounting for approximately 45% of the total particle weight at a diameter of 0.6 mm. Furthermore, sieve number 16 and 50 retained 24% and 21% of the mass, respectively. This implies that the particle size of the C-S particles ranged from 0.3 mm to 1.18 mm, with a significant concentration of particles at the 0.6 mm size.

While the outcomes of C-C from sieve analysis illustrated in table 2 revealed that 56 % of the particles at size 1.18 mm. However, particles size at 0.6 and 2 mm achieved 20% from the total weight of the tested sample for each one. However, the range of the particles size for C-C considered

the widest in comparison with C-S and the largest amount of the dominant particle size is 0.6 to 2 mm.

Although, there are different amount from the sample retained on sieves 50, 30, 16 and 10. When the sieve analysis conducted for Bio-A, it is obvious the largest amount of particles retained on sieve 10. Then quantity of Bio-A' sample decreased sequentially for the sieves 50, 30 and 16 at percentage 8.5, 12 and 28% respectively. The final results showed that the particle sizes for Bio-A offers a wider range than the particle sizes of C-S and C-C.

Table 3. Sieve analysis outcomes for Bio-A.

Sieve No.	4	6	10	16	30	50	Pan
Diameter mm	4.7	3.35	2	1.18	0.6	0.3	
Retained mass g	0	14	304.5	196	84	59.5	42
Retained %	0	2	43.5	28	12	8.5	6
Cumu. retained %	0	2	45.5	73.5	85.5	94	100
Finer %	100	98	54.5	26.5	14.5	6	0

The sieve analysis has revealed an outcomes, which assist to identify the particles' size and the particles' size range, consequently, specify for each material the surface area of its particles. The surface area of the particle is a vital issue in determining the reaction rate between the impurities and the filter media. The particles have small diameter is exhibit large surface area in comparison with particles have larger diameter. Hence, if an equal quantity of all tested material packed in the filter system, this will lead to give an insight for the sequence of the filter media according to their surface area from the largest to the smallest.
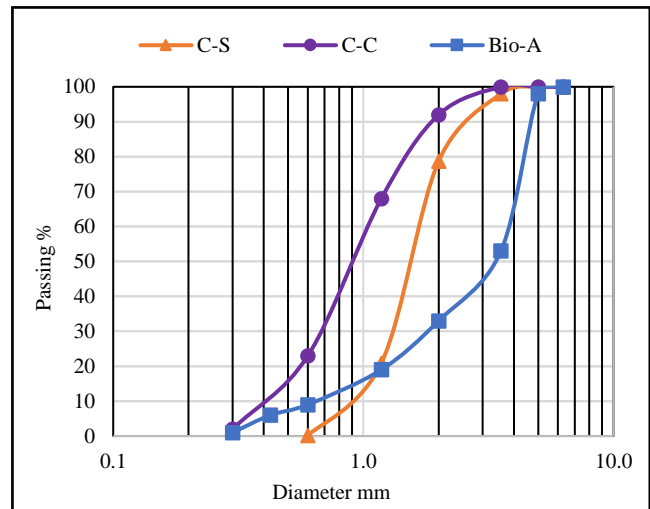

Figure 1. Particle size distribution curve PSD

Based on the findings of the sieve analysis that shown in tables 1, 2 and 3 the sequence of materials according to surface area is (C-S > C-C > Bio-A). PSD curves for the results of the sieve analysis were created as illustrate in Fig. 1. The curves assisted to estimate the main parameters for the tested materials such as uniformity coefficient of gradation C_c and coefficient UC. The values of d10, d30 and d60 were obtained from the PSD curves and then applied in equation 1 and 2 as shown below to determine the UC and C_G.

$$UC = \frac{d60}{d10} \tag{1}$$

$$C_c = \frac{d30^2}{d10 \times d60} \tag{2}$$

Table 4 present the values of C_c and UC. Both parameters are required as a method of validation for selected materials in this research. The determined UC of the sample which its value more than 4 and value of C_c in the range between 1 and 3 is described as a well graded material. The values of UC for C-S and C-C are below 4 and the C_c in range 1 to 3; this indicate both material are categorized as uniformly graded and poorly graded. However, the value of UC for Bio-A is between 4 and 6 and the Cc in range 1 to 3; this refer to Bio-A as a well graded material (mass included vast variety sizes for particle). The description of the results for selected materials according to PSD, some tested samples indicate higher surface area.

Table 4. The main parameters of PSD curves.

Material		C-S	C-C	Bio-A
	d10	0.43	0.68	0.68
Effective Size	d30	0.78	1.44	1.82
	d60	1.06	2.12	3.84
Uniformity Coefficient	UC	2.45	3.12	5.67
Coefficient of Gradation	C_c	1.34	1.44	1.27

3.1.2. Specific gravity S.G

Washing is an essential operation in filtration system that cause a dynamic motion for the material that act as filtration media. Consequently, the media of the filters should not be contained very light particles to prevent them from floating.

Table 5. Physical characteristics for tested material.

Material	C-S	C-C	Bio-A
Specific Gravity S.G	2.57	2.65	1.17
Porosity ε	0.36	0.46	0.73
Bulk density g/cm³	1.58	1.47	0.41
Hydraulic conductivity m/s	0.11	0.014	0.029

Therefore, analysis of specific gravity for the selected material was achieved as illustrated in table 5, and it is noticeable that the specific gravity of Bio-A is 1.17. Even under low flow rate, the materials with specific gravity less than 1 are exposed for floating. In case of high flow rate for the effluent the possibility of floating is high for Bio-A. On the other hand, C-S and C-C presented specific gravity 2.57 and 2.65 respectively. This results showed an advantage for C-S and C-C over Bio-A in term of physical characteristics when they utilize as filter media.

3.1.3. Porosity

Porosity is represent the fraction ratio for the volume of the voids in respect to the sample' total volume. The range of the porosity' values are from 0 to 1. Based on the result analysis for sieve test two materials' groups were obtained. First group, included the material which classified as well graded like Bio-A, and the second one is the material which described as uniformly graded such as C-C and C-S. It is ordinary that the materials have lowest porosity tend to be well-graded materials in comparison with uniformly graded materials due to that the particles with small size can fill the pores effectively. However, the outcomes which is obtained from the test of the porosity presented a different expectation. The measured porosity for Bio-A was 0.73; the measured porosity for C-S and C-C were 0.36 and 0.46. Irregular shape for the surfaces of Bio-A particles and the many pores and bends lead to believe that Bio-A has large surface area.

3.1.4. Bulk density

Bulk density is one of the physical element which assist in term of understanding the particles behaviour of the media inside the filtration systems. Simply, the Bulk density can be defined as the dry weight of the sample over the volume that includes the whole particles even the space of the pores. Washing system exposed the media inside the filtration systems for a dynamic motion. Consequently, identification the bulk density value play significant role to maintain the media' motion within the filtration system. Cheremisinoff [21] states the packing density is an identification for bulk density. Cheremisinoff [21] indicates that the materials can be categorized based on their measures bulk density. According to the literature review, he listed three categories for bulk density as illustrated in table 6. The data which is illustrated can be considered as a validation indicator to get an insight about the materials' appropriateness to behave as filter media. Bulk density for C-S and C-C were 1580 and 1470 kg/m³ respectively. On the other hand, the bulk density rating less than 600 for the Bio-A, its considered as a light material.

Table 6. Bulk Density categories.

Materials characterization	Light	Average	Extra heavy
Bulk density kg/m³	< 600	600 < Bd < 2000	> 2000

3.2. Chemical characteristics

The chemical composition of the selected materials was performed according to X-ray fluorescence spectrometer XRF as illustrated in table 7. The primary goal of this research is to identify the composition of materials; if they are contain oxides that have tendency to retain the ions of phosphate such as CaO, Al_2O_3, MgO, and Fe_2O_3. In addition, to make sure that the tested materials are free from oxide that classified as a hazardous oxides such as heavy metals. The findings showed that the calcium oxide available with significant portion in C-C, which revealed at least 84.16 % from the total composition. Furthermore, C-C is free from the oxides such as Fe_2O_3 and Al_2O_3 and only contain minor portion of MgO. Hence, the dominant oxide that revealed by XRF for chemical composition of C-C is CaO. On the other hand, the Bio-A contains a minor portions of MgO and CaO in comparison with C-C as showed in table.7. However, the Bio-A consist of

reasonable amount of Al_2O_3 and Fe_2O_3. It is obvious that the Bio-A consists of 4.39 % of Fe_2O_3 and 7.16 % of Al_2O_3.

Table 7. Analysis of X-ray fluorescence.

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
C-C	14.846	. . . .	. . . .	84.162	0.513	. . . .
Bio-A	12.493	7.168	4.397	7.615	0.451	0.181
Oxides	P_2O_5	SrO	MnO	CHO	ZnO	CuO
C-C	0.25	0.031	. . . .	. . . .	0.003	0.005
Bio-A	0.512	0.012	0.103	63.957	0.005	0.004

According to the literature review from the previous section and the outcomes from table 7, it is anticipated that the tested materials tend to be phosphate removal media. However, the removal degree will be different based on the quantity and sort of components which formed the materials' chemical composition. It is noticeable that the C-S' chemical composition is not tabulated in table 7; the justifications is based on the previous studies for the C-S' XRF analysis which they illustrated that the dominant component in C-S formation is silica.

Oxides that shown in table 7, such as ZrO_2, ZnO and CuO indicate the presence of heavy metals. Obviously, they are represent a minor quantities as components in the selected materials' composition. This results offered an insight, that the potential leaching from materials' solution is secure.

3.3. Batch test results

A comparison was conducted for three selected materials based on their capability for phosphate removal as illustrated in figure 2a, 2b and 2c, the outcomes were obtained from batch experiments. Obviously, over all the experiments intervals the Bio-A presented the maximum phosphate removal as shown in bar charts. Whereas the phosphate removal capability for materials such as C-C and C-S exhibit the minimum in comparison with Bio-A. However, C-C indicates better removal efficiency than C-S. The measurement for removal the phosphate is expressed by percentages.

In general, it is noticeable that the removal quantities were correlate to the experiments time (experiments time divided into three figures for clarity) at similar pattern. The first sample was grabbed after 5 min from the experiment running. The results showed a dropping in the concentrations of the phosphate to 33, 19.17 and 33.50 % for the C-C, C-S and Bio-A, respectively. During the subsequent experiment intervals, the rate of phosphate removal became slower over the experiments duration as shown in figures 2b and 2c. The overall time of experiments conduction were 24 hours. At the end of the batch test, the ultimate quantity for phosphate removal by Bio-A as percentage was 70 %. On the other hand, the C-C and C-S accomplished removal quantities at 55.10 and 34.58 %, respectively. According to the batch experiments outcomes the Bio-A, more investigations about it is significance for phosphate sorption are required. However, Bio-A has poor physical characteristics comparatively to the C-C and C-S. While these materials have low affinity to the phosphate and good physical characteristics. The outcomes that obtained from batch test indicated that the materials that contain oxides such as Al_2O_3 and Fe_2O_3 like Bio-A exhibited good performance in comparison with material consisting of oxides of CaO such as C-C. It is possible that the retention mechanism for phosphate is playing a crucial role in improving the removal efficiency. Ion exchange is the mechanism that lead to adsorb the phosphate on the tested materials' surfaces which they consist of Fe/Al oxides. Whereas the rich materials with soluble Ca, the removal process occur via formation of

Ca-phosphate precipitate. It is anticipated that the performance of phosphate removal via filter media contain Fe/Al oxides considered more adequate than reducing the phosphate via formation of Ca-phosphate precipitate (the other factors were excluded). According to the batch experiment outcomes the removal pattern can be described as follow: at first the phosphate concentrations for materials contain Fe/Al oxides were decreased faster than the end of the experiments. In contrast, slow rate of phosphate removal was occur via rich Ca material. Consequently, the rate increased in later intervals. Although, Bio-A presented an outstanding performance for phosphate removal.
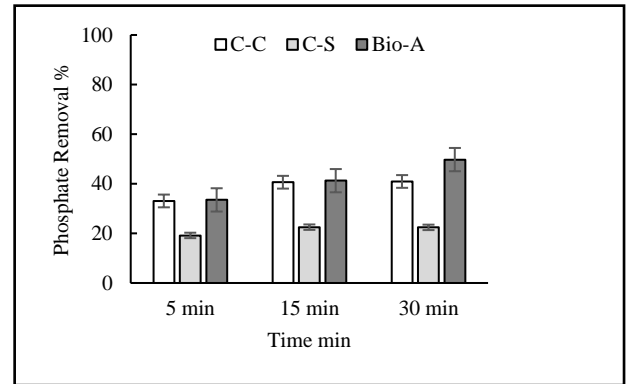
a. Time interval (0 to 30 min)
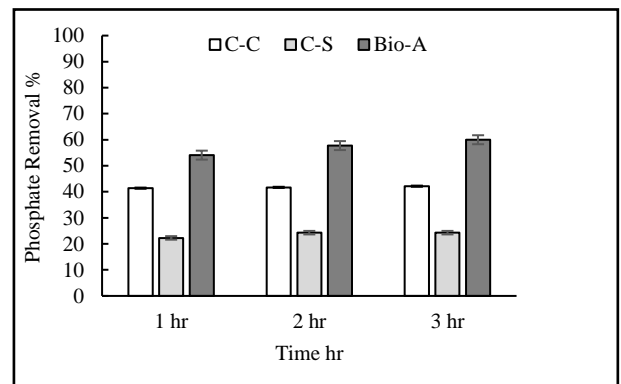
b. Time interval (30 min to 3 hr)
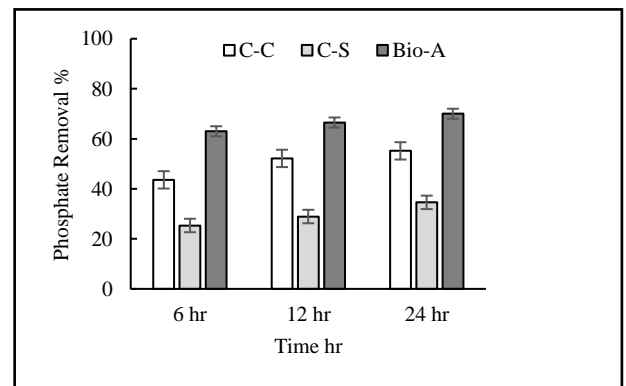
C. Time interval (3 hr to 24 hr)
Figure 2. Batch experiments results

4. Conclusion

In conclusion, the study concludes that Bio-A are highly efficient material for phosphate removal, while C-C and C-S exhibit weak performance in comparison. Investigation the characteristics for filter media is the initial part of the research that was executed to identify the selected materials' suitability for the filtration systems. C-C and C-S were exhibit a significant physical characteristics as a packed filter media in comparison with Bio-A. On the other hand, Bio-A presented acceptable contain of Fe/Al hydroxides. This lead to conclude that the selection method for filter materials is grounded on that the majority of materials which they presented adequate phosphate sorption have a tendency to contain easily soluble Ca/Mg compounds or Fe/Al hydroxides. The outstanding phosphate removal by Bio-A, combined with the favorable physical properties of C-C and C-S, leads to the decision to employ coating technology. Bio-A will serve as the coating dosage, covering the support media (C-S and C-S). Bio-A will provide phosphate removal capabilities, while C-C and C-S will provide the support media (according to their physical properties) for the newly created filter material. This coating approach presents a potential solution to optimize the overall performance of filters for phosphate removal.

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