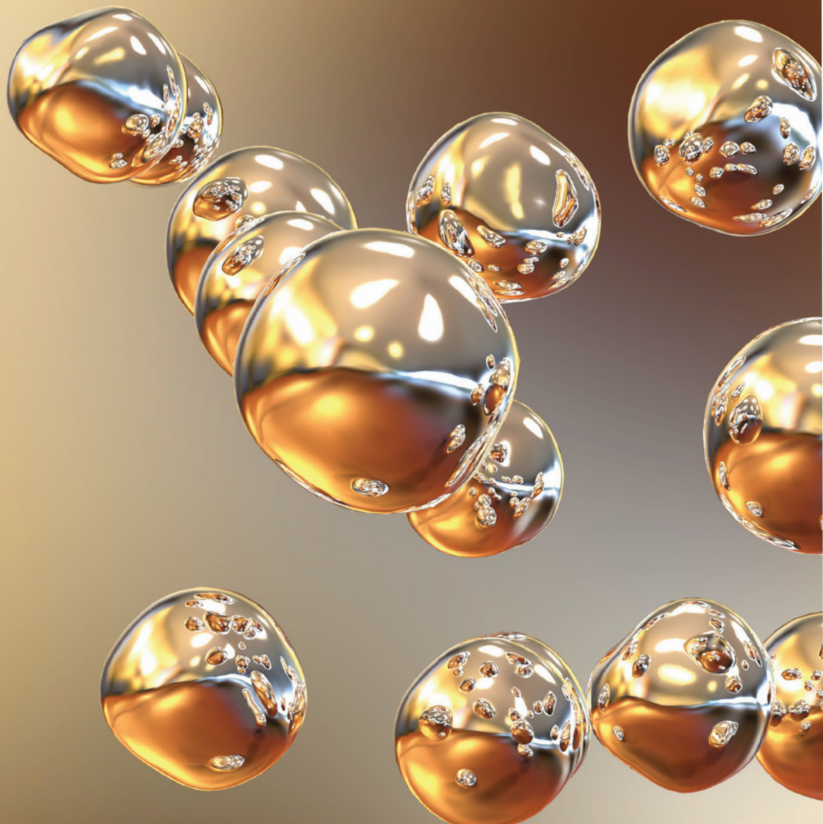
THE GREAT WORLD OF NANOTECHNOLOGY



Marcos Augusto de Lima Nobre

(Organizador)

VOL II

EDITORA ARTEMIS

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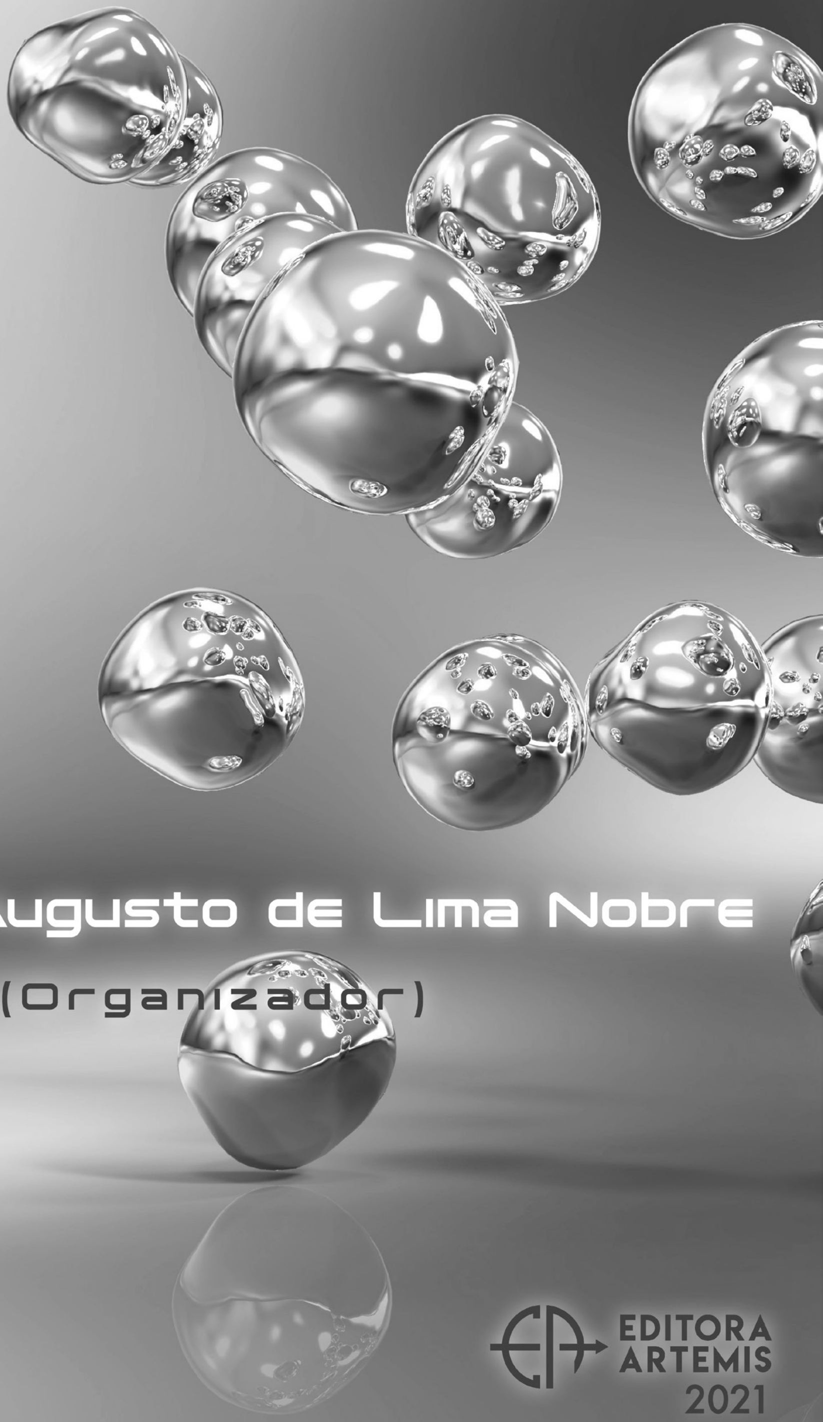

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PREFACE

The insertion of new and enhanced materials based on materials belonging to the Nano scale in the day-by-day has growth up in a silent way. In part, a number of works in the nanotechnology stemming of theoretical research using Density Functional Theory (DFT) and sophisticated simulation methods; another part is associated to the protected technologies associated to the military and patented nanomaterial and its process. In this sense, open access to recent aspects on the nanostructures application and properties can be reached in this book. Here, an interesting set of chapters gives opportunity of access texts that reach process and processing of nanostructures, applications of nanotechnology, advanced techniques to theoretical development. A broad set of nanostructures are here covered such as, nanocrystal, superficial nanograins, inner microstructures with nanograins, nanoaggregates, nanoshells, nanotubes, nanoflowers, nanoroad, nanosheets, Also, reveals new investigations areas as grainboundary of nanograins in ceramics and metals. A great number of software has been used as a tool of development of Science and Technologies for nanotechnology COMSOL Multiphysics 5.2. Phenomena and properties has been investigated by recent or classical techniques of materials characterization as Localized Surface Plasmon Resonance (LSPR), X-ray photoelectron spectroscopy (XPS), Field Emission Gun Scanning Electron Microscopy (FEG-SEM) with Energy Dispersive Spectroscopy (EDS), Raman Scattering Spectroscopy (RSS), X ray diffraction (XRD), ^{57}Fe Mössbauer spectroscopy, UV-vis spectroscopy, dynamic light scattering (DLS), Atomic Force Microscopy (AFM), and Field Emission Gun Scanning Electron Microscopy (FEG-SEM). In this sense, collections of spectra from Mössbauer spectroscopy, UV-vis spectroscopy and Infrared spectroscopy can be found. As a matter of fact, some chapter's item can be seemed as specific protocols for synthesis, preparations and measurements in the nanotechnology.

I hope you enjoy your reading.

Prof. Dr. Marcos Augusto Lima Nobre

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CHAPTER 9

APPLICATION OF CLAY-CARBOXIMETHYLCHITOSANE NANOCOMPOSITE-SILVER NANOPARTICLES IN FILTERS TO TREAT CONSUMPTION WATER IN RURAL AREAS OF CAMANA - AREQUIPA-PERU

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ABSTRACT: A filter was formulated with a clay nanocomposite-carboxymethylchitosan-silver nanoparticles, which was used to eliminate bacteria and anions in water for human consumption. Silver nanoparticles were obtained by reduction of 1mM silver nitrate, with 0.01% carboxymethylchitosan and 2mM sodium borohydride as initiator of the reduction. Subsequently, the filters were obtained as containers with clay and wood sawdust as pore former, in the proportion of 2: 1; it was molded, dried and sintered at 800 ° C for 3 hours. The filters were then impregnated with the colloidal dispersion of carboxymethylchitosan-silver nanoparticles, by immersion for 12 hours. Four water sampling points were defined: Huacapuy, La Punta, Plaza de Quilca, Caleta de Quilca. When evaluating the filtering capacity of the filters, it was found that they are removed: conductivity 84.03% in La Plaza de Quilca, sulfates 85.48% in La Plaza de Quilca; Chlorides 93.93% in La Plaza de Quilca. On the other hand, fecal coliform and total coliform bacteria were 100% removed in La Punta and in the Plaza de Quilca. Coliform bacteria were not found in Huacapuy or in Caleta de Quilca. Therefore, it can be confirmed that the results have a significant removal percentage in the elimination of anions, in terms of total coliform bacteria and fecal coliforms they were completely removed.

KEYWORDS: Nanocomposite. Clay. Filter. Carboxymethylchitosan. Silver nanoparticles.

1 INTRODUCTION

Nanotechnology is the study, design, creation, synthesis, manipulation and application of materials, devices and functional systems through the control of matter at the nanoscale, and the exploitation of phenomena and properties of matter at the nanoscale, at this scale and molecules, show totally new phenomena and properties. Therefore, scientists use nanotechnology to design novel and inexpensive materials with unique properties [1].

These phenomena are governed under the laws of colloidal thermodynamics and their properties are between quantum mechanical phenomena and classical mechanics, these new properties are what scientists take advantage of to synthesize new materials (nanomaterials) or nanotechnological devices, in this way Nanotechnology promises solutions to multiple problems that humanity currently faces such as: environmental, energy, health (nano medicine), and many others, however these new technologies can lead to risks and dangers if they are misused [2].

Nanomaterials are a new class of materials, whether they are ceramics, metals, semiconductors, polymers or a combination of these or nanocomposites, in which at least one of its components has one of its dimensions between 1 and 100 nm; they represent a transition between molecules, atoms and a material with dimensions of volumetric solid. Due to its size reduced to nanometers, the physicochemical properties differ for the same material, molecules and atoms [3].

Treatment of drinking water is a viable option to improve and ensure water quality, mainly in places that do not have treatment systems, or existing systems operate poorly or there are drought conditions, in the comparative study of two filtration systems for drinking water at home[4], two systems were evaluated: a filter with 1 candle (1VC) and another with 2 ceramic candles (2VC) for 6 months. The efficiency of turbidity reduction and E. coli was evaluated. A synthetic substrate was used whose average turbidity value was 32.7 ± 2.81 NTU and 3.9x105 CFU / 100mL of E. coli. The results showed that both filtration systems were able to reduce turbidity to average values of 0.28 NTU (99% efficiency) and eliminate E. coli between 99.99 and 100%. No statistically significant differences were found in terms of the quality of water filtered by both systems.

A conventional method is applied to produce porous ceramics, by addition and pyrolysis of an organic material: starch, which acts as a pore former. The aqueous suspensions of (52-55 V%) of the zirconium mixture were stabilized with a commercial ammonium polyacrylate solution as dispersant and consolidated in plastic molds at 90 ° C for 30 minutes. Then they were sintered between 1000-1500 ° C for 2 h. The characteristics of the product were evaluated by measurements of density, volumetric contraction, mercury intrusion and the evolution of the crystalline phases by X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was found that the microstructural properties of the ceramic (pore volume, relationship between open and closed porosity, size distribution, pore morphology) depend on the amount of starch added and the sintering temperature [5]

2 EXPERIMENTAL PART

The experimental development was carried out in the Laboratories of the Academic Department of Chemistry of the San Agustin de Arequipa National University.

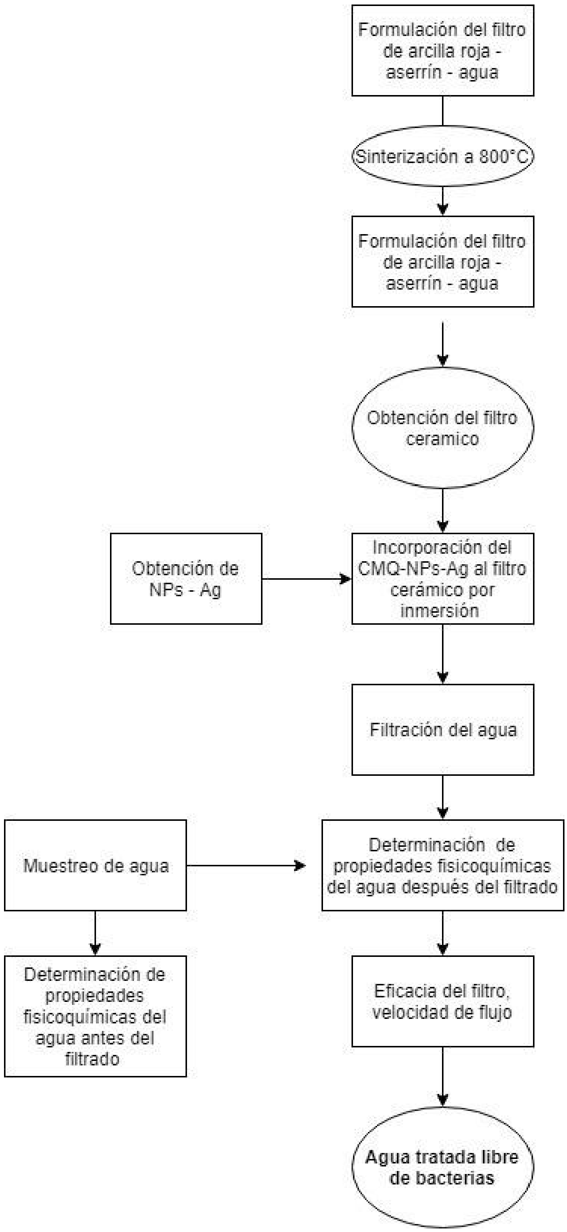
The clay samples, used as the matrix of the nanocomposite, were sampled in the Yarabamba district, which is located southwest of the city of Arequipa, whose coordinates are: Latitude: -16.5481, Longitude: -71.4775; 16 ° 32 ′ 53 ″ South, 71 ° 28 ′ 39 ″ West, were placed in hermetic plastic bags and were transported to the laboratory for characterization. Regarding the structure and composition of the clay, it was carried out by scanning electron microscopy techniques (SEM - EDX), in the physicochemical characterization the following parameters were determined: pH, soluble salts, exchangeable bases, cation exchange capacity (CEC) and absolute density [6].

The obtaining of silver nanoparticles was by chemical reduction of silver nitrate with carboxymethylchitosan using 2mM sodium borohydride as initiator of the reduction; the evaluation of the plasmon of the silver nanoparticles was carried out by UV-visible spectroscopy and dynamic light scattering (DLS) [7].

Filters were obtained by making a mixture of clay-sawdust (as pore former, in the ratio 2: 1) and water to form a paste, molded, dried and sintered at 800 ° C for 3 hours. Subsequently, the filters were treated by the immersion method in the colloidal dispersion of silver-carboxymethyl chitosan nanoparticles for 12 hours, they were dried and with these filters the tests of the water under study were carried out [10].

In water for human consumption, the following variables were measured: pH, conductivity, turbidity, chlorides, sulfates, fluorides, nitrates, nitrites, phosphates and the count of microorganisms; numbering of total coliforms, numbering of fecal coliforms (NMP), by AWWA methods, before and after filtering [8] and in this way the efficiency of the filter was determined. The water sampling points were: Huacapuy, La Punta, Plaza de Quilca, Caleta de Quilca, located in Camaná located in the south western part of Arequipa, whose coordinates are: Latitude: -16.6238, Longitude: -72.7105; 16 ° 37 ′ 26 ″ South, 72 ° 42 ′ 38 ″ West. The tests were carried out with the water before filtering, with the water passed through the filter impregnated with the colloidal dispersion of the nanocomposite: carboxymethylchitosan-silver nanoparticles. Figure N° 1 shows the block diagram of the process for obtaining and operating the filters with the nanocomposite clay-Carboxymethylchitosan-Silver Nanoparticles.

Figure N° 1: Block Diagram of the obtaining and operation of the filter with the clay nanocomposite-Carboxymethylchitosan-Silver Nanoparticles.



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3 RESULTS AND DISCUSSION

3.1 SYNTHESIS OF SILVER NANOPARTICLES:

The synthesis was made by chemical reduction of 1mM [9] silver nitrate with carboxymethylchitosan and 2mM sodium borohydride as reaction precursor, according to the following equation:

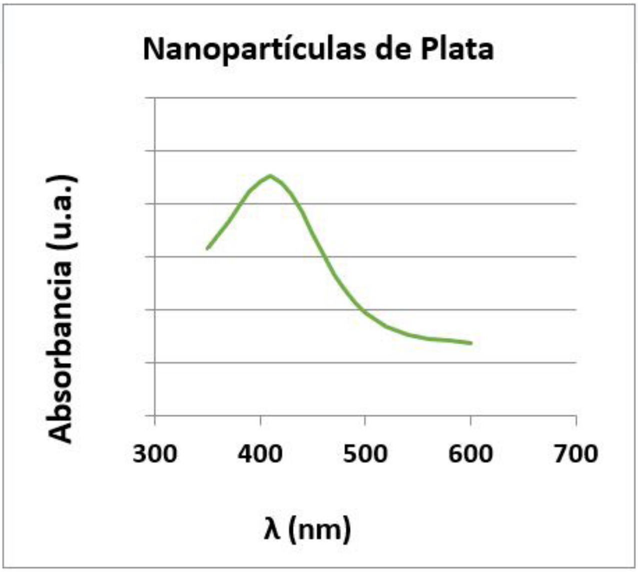
$$4\ AgNO_3 + NaBH_4 + 3H_2O \rightarrow 4Ag + H_3BO_3 + 3HNO_3 + NaNO_3 + 2H_2$$

When performing the synthesis, a yellow solution was observed due to the resonance of the plasmon of the silver nanoparticles, which indicates its presence.

3.2 EVALUATION OF SILVER NANOPARTICLES BY UV-VISIBLE SPECTROSCOPY

When characterizing the nanoparticles by UV-visible, it was found that the maximum absorbance is between 410 nm and 420 nm, which is a characteristic of the presence of spherical silver nanoparticles. Metals such as silver, which have free electrons, show a resonance plasmon in the visible spectrum, giving rise to colors not observed in the same materials on a macro scale [11]. For example, silver nanoparticles show an intense SPR (surface plasmon resonance) mainly in the wavelength ranges of 410-420 nm, as seen in Figure N° 2:

Figure N° 2: UV-visible spectrum of silver nanoparticles



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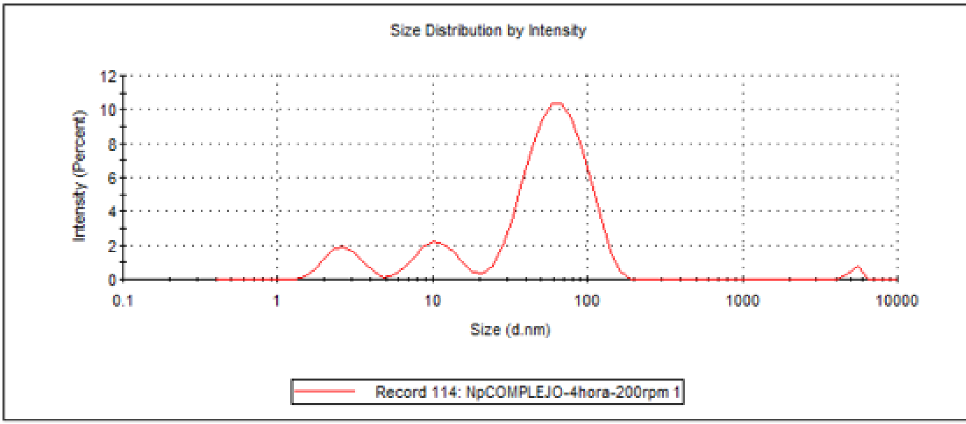
An absorbance maximum is observed between 410 nm and 420 nm, which is a characteristic of the presence of silver nanoparticles, due to the resonance of surface plasmons.

3.3 SIZE EVALUATION OF SILVER NANOPARTICLES BY DYNAMIC LIGHT SCATTERING (DLS)

In the evaluation by Dynamic Light Scattering (DLS), the hydrodynamic diameter of the silver nanoparticles was determined. In the distribution histograms, there are sizes of 2,716 nm, 11.05 and 67.63nm; what is observed in Figure N° 3:

Figure N° 3: Graph of the histogram of the hydrodynamic diameters of the silver Nanoparticles by Dynamic Light Scattering (DLS)



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In the histogram of the hydrodynamic diameters of the silver nanoparticles, a polydisperse distribution is observed with sizes of hydrodynamic diameter reached 2,716 nm in 8.7%; 11.05nm by 11.8% and 67.63nm by 78.4%; size less than 100 nm, which confirms the presence of nanoparticles.

3.4 PHYSICOCHEMICAL CHARACTERIZATION OF THE CLAY

Below in Table N° 1 are the results of the physicochemical properties of the clay:

Table N° 1: Physicochemical Characterization of the clay

Sample	**pH**	**Soluble salts g/%**	**Interchangeable bases %**	**C I C (Cation exchange capacity)** meq/100g	**Density g/cm³**	**Humidity %**	**Carbonates %**
Clay	5,6	0,06	27,84	16,60	1,595	3,608	0,00

Source: self made

When characterizing the clay, it is found that it has a high value of exchangeable bases: 27.84%, as well as a high value of Ion Exchange Capacity (CEC) of 16.60 meq / 100g, so it would be easy for these bases to come out or cations, creating free spaces.

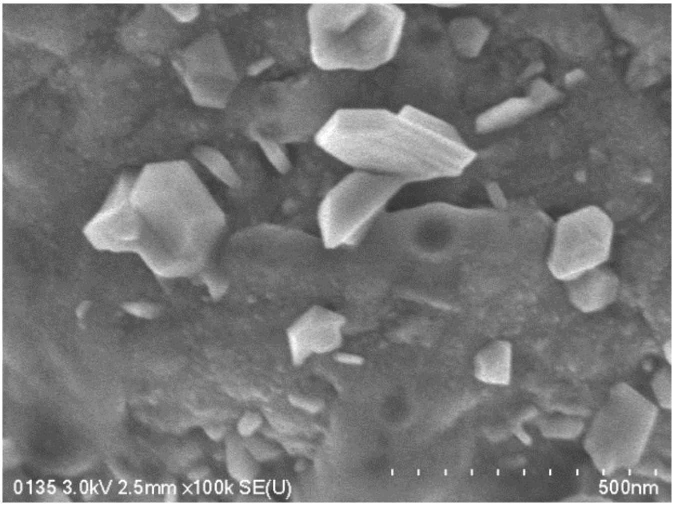
3.5 OBTAINING THE CLAY-SAWDUST FILTER

The clay-sawdust filters were obtained by mixing 100 mesh pulverized clay and 100 mesh pulverized wood sawdust, in a 2: 1 ratio with water, they were molded, dried and brought to a temperature of 800°C, obtaining a hard ceramic of russet. Porosity is an important property of a ceramic filter, since the water filtration flow depends on it, this porosity is obtained by the sawdust present in the mixture and occurs when the filter is subjected to temperatures above 800°C.

3.6 MORPHOLOGICAL CHARACTERIZATION BY SEM-EDX OF THE CERAMIC OBTAINED

The micrograph of the ceramic obtained is shown below, in Figure N° 4:

Figure N° 4: Micrograph of the ceramic



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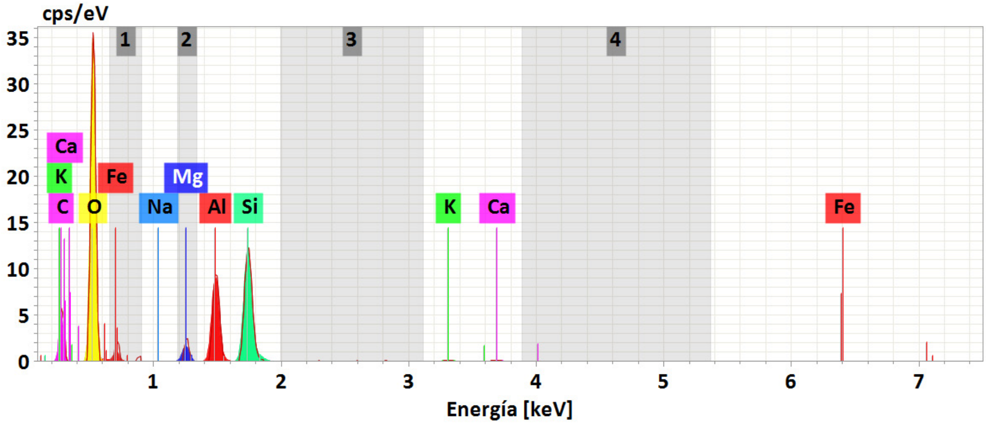
The micrography taken by SEM shows a magnification of the surfaces of 100 thousand times, the structure of the ceramic is observed, with crystallizations corresponding to the typical morphology of clay in sizes from 50 nm to 200 nm. Likewise, an inhomogeneous phase with small depressions is observed. The most dominant phase are homogeneous surfaces.

We can also observe the spectrum of the results of the elemental microanalysis of the ceramic obtained by EDX, in Figure N° 5:

Figure N° 5: Elemental microanalysis of the ceramic obtained by EDX



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In this spectrum it can be observed, different signals that show a higher proportion of oxygen, silicon, followed by aluminum, carbon and iron and to a lesser extent as minorities: potassium, calcium, magnesium and sodium.

The general composition indicates that there is a high amount of silicon and oxygen (silicates), minor amounts of calcium carbonate (calcite), and probably small fractions of aluminum and magnesium silicates in the form of micas or feldspar and iron oxides.

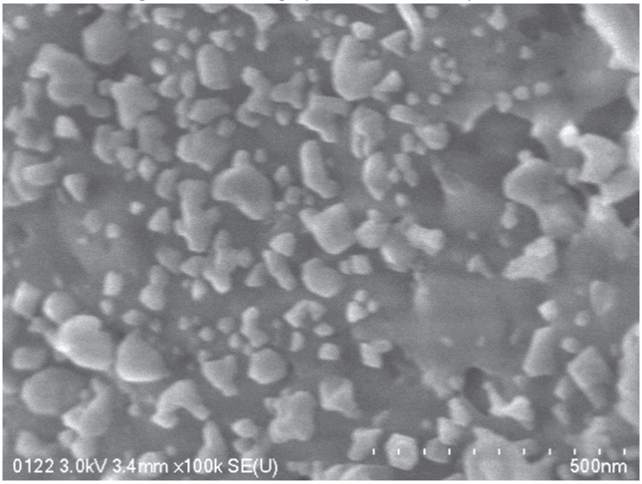
3.7 IMMERSION PROCESS OF THE FILTER IN THE COLLOIDAL DISPERSION OF CARBOXYMETHYLCHITOSAN-SILVER NANOPARTICLES

The nanoparticles were incorporated into the filter by the in situ immersion method, which consists of immersing the filter in the silver-carboxymethyl chitosan nanoparticle solution for 12 hours, they were dried at room temperature and the tests were carried out with these filters.

3.8 MORPHOLOGICAL CHARACTERIZATION BY SEM-EDX OF THE NANOCOMPOSITE: CERAMIC - CARBOXYMETHYLCHITOSAN - SILVER NANOPARTICLES

Below is a micrograph of the nanocomposite: carboxymethylchitosan ceramic-silver nanoparticles, in Figure N° 6:

Figure N° 6: Micrograph of the nanocomposite



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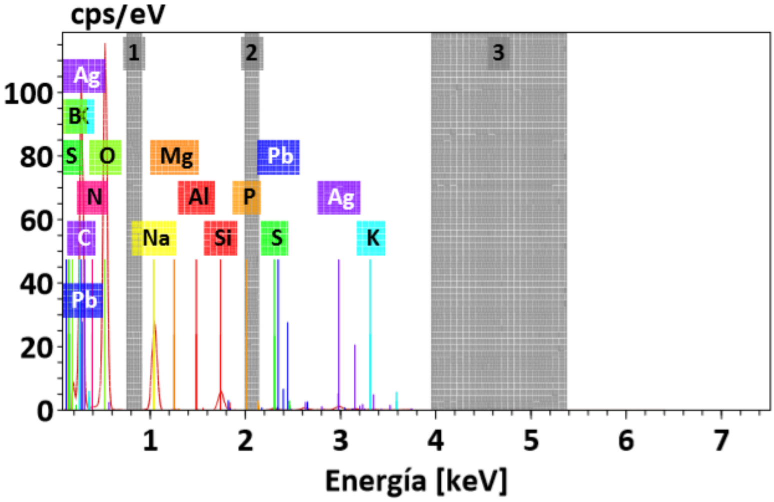
The micrograph taken by SEM shows a magnification of the surfaces of 100 thousand times, crystallizations corresponding to the typical morphology of clay are observed, with sizes between 20 nm and 100 nm and a layer that covers the crystallizations, which would correspond to carboxymethylchitosan with nanoparticles silver.

We can also observe the spectrum of the results of the elemental microanalysis of the ceramic obtained by EDX, in Figure N° 7:

Figure N° 7: EDX analysis of the ceramic obtained



Source: *self made*

In this spectrum it can be observed, different signals that show a higher proportion of oxygen, sodium, silicon, sulfur followed by carbon, boron, nitrogen, aluminum and to a lesser extent as minorities: potassium, magnesium and the presence of silver is notorious due to the silver nanoparticles.

3.9 PHYSICOCHEMICAL AND BACTERIOLOGICAL PROPERTIES OF THE WATERS UNDER STUDY BEFORE AND AFTER FILTERING.

To carry out this step, the waters under study were filtered with the ceramic filter obtained and the following physicochemical tests were carried out: pH, conductivity, turbidity, anions [12], as well as the bactericidal properties of the nanocomposite, determining the count of microorganisms: Numbering of Total Coliforms, Fecal Coliform Numbering, using AWWA [13] Standard Methods. The tests were carried out on the water samples before and after filtering. Table N° 2 is shown below with the results obtained.

TABLE N° 2: Water quality versus the physicochemical and bacteriological values before and after filtering.

Variables	**Normatividad (SUNASS)**	**Huacapuy**			**La Punta**			**Plaza de Quilca**			**Caleta de Quilca**		
		Ant Filt	Desp Filt	% Remoc	Ant Filt	Desp Filt	% Remoc	Ant Filt	Desp Filt	% Remoc	Ant Filt	Desp Filt	% Remoc
pH (0 a 14)	6,5 a 8,5	7,8	7,7	0,1	7,8	7,6	0,2	7,9	7.9	0	7,8	7,7	0,1
Conductiv (µS)	1500	1180	520	55,93	1590	570	64,15	5260	840	84,03	5050	872	82,73
Turbiedad (NTU)	5	0,72	0,43	40,28	0,7	0,36	48,87	0,8	0.29	63,65	0,64	0,3	53,12
Cl- mg/L	250	112	96,4	13,93	146,3	102,56	29,75	1344,6	81.57	93,93	1356,67	87,05	93,58
$(SO_4)^{2-}$ (mg/L)	250	236,32	156,45	33,80	345,2	205,2	40,56	1611,6	234.9	85,42	1621,4	249,6	84,60
F- (mg/L)	1,0	0,43	0,41	4,65	0,46	0,44	4,35	0,48	0.47	2,08	0,48	0,46	4,17
$(NO_3)^-$ (mg/L)	50	4,83	4,81	0,41	10,95	10,91	0,37	38,29	15.72	58,94	37,27	13,86	62,81
$(NO_2)^-$ (mg/L)	0,2	< 0,002	< 0,002	_	< 0,002	< 0,002	_	< 0,002	< 0.002	_	< 0,002	< 0,002	–
$(PO_4)^{3-}$ (mg/L)	0,4	0,21	0,19	9,52	0,16	0,13	18,75	0,07	< 0.002	100	0,17	< 0,002	100
C.F. (UFC/100ml)	0 (ausencia)	< 1,1	< 1,1	_	11	< 1,1	100	92	< 1.1	100	< 1,1	< 1,1	_
C.T. (UFC/100ml)	0 (ausencia)	< 1,1	< 1,1	_	1,6	< 1,1	100	2,2	< 1.1	100	< 1,1	< 1,1	_

Source: *self made*

To do the analysis of the results, Table N° 3 was made where the values before filtering are appreciated, which exceeded the values allowed by the National Superintendency of Sanitation Services (SUNASS) and that after filtering they decreased to normal values:

TABLE N° 3: Summary table of the physicochemical and bacteriological values that exceeded the established values before and after filtering.

Variables	**Normativity (SUNASS)**	**Huacapuy**		**La Punta**		**Plaza de Quilca**		**Caleta de Quilca**	
		Before Filter	After Filter	Before Filter	After Filter	Before Filter	After Filter	Before Filter	After Filter
Conductiv (µS)	1500	1180	520	1590	570	5260	840	5050	872
Cl- mg/L	250	112	96,4	146,3	102,56	1344,6	81,57	1356,67	87,05
$(SO_4)^{2-}$ (mg/L)	250	236,32	156,45	345,2	205,2	1611,6	234,9	1621,4	246,6
C.F.(UFC/100ml)	0 (absence)	< 1,1	< 1,1	11	< 1,1	92	< 1,1	< 1,1	< 1,1
C.T.(UFC/100ml)	0 (absence)	< 1,1	< 1,1	1,6	< 1,1	2,2	< 1,1	< 1,1	< 1,1

Source: self made

In Table N° 3, it is observed that comparing the values established according to (SUNASS) with the values found in the different sampling points, it is found that: the waters after filtering by applying the nanocomposite, it was found that they remove the following in a greater proportion values: conductivity: 84.03% in La Plaza de Quilca, sulfates 85.42% in La Plaza de Quilca, chlorides 93.93% in La Plaza de Quilca. On the other hand, the Fecal Coliform and Total Coliform bacteria were 100% removed in La Punta and in the Plaza de Quilca. Coliform bacteria were not found in Huacapuy or in Caleta de Quilca. Therefore, it can be confirmed that the results have a significant removal percentage in the removal of anions and in terms of total coliform bacteria and fecal coliforms as a whole.

4 FILTER OPERATION

The procedure is based on microfiltration through a porous material, where the filter pores have a size between 0.6 and 0.3 µm, approximately determined by SEM. According to Van Der L.H. [15], if the pore size would be 0.1 µm, the filter would not need a disinfectant for the elimination of Escherichia coli, which has a size between 0.5 to 1 µm.

On the other hand, according to Ludeña J. [14], the presence of silver nanoparticles near a virus, fungus, bacterium or any other unicellular pathogenic microbe, incapacitates the oxygen metabolism enzyme and in a few minutes the pathogenic microbe suffocates, dies and It is eliminated from the body by the immune and lymphatic systems.

Vidal S.10 indicates that if silver binds to the cell membrane of bacteria, they increase their size and cytoplasmic content and present abnormalities that result in cell lysis and death.

According to the results obtained from Lantagne, the clay and sawdust-based filters form pores of 0.6 to 3 μm, which was determined by SEM, and microorganisms such as parasites are eliminated, including Giardia and Cryptosporidium, ranging from 5 - 7 μm and 5 μm, respectively. 99.99% but not 100% so it is necessary to impregnate the colloidal silver as it completely removes the bacteria.

5 CONCLUSIONS

When applying to the water samples under study, the filtering system based on the clay-carboxymethylchitosan nanocomposite - silver nanoparticles, the quality of the waters contaminated with bacteria and chemical substances was improved, producing water suitable for human consumption, therefore a low-cost filter system, simple technology, ecologically acceptable and economically accessible to low-income rural populations is being developed.

After filtering the waters using the nanocomposite, it was found that they remove the following values in a greater proportion: conductivity: 84.03% in La Plaza de Quilca, sulfates 85.42% in La Plaza de Quilca, chlorides 93.93% in Quilca Square. On the other hand, the Fecal Coliform and Total Coliform bacteria were 100% removed in La Punta and in the Plaza de Quilca. Coliform bacteria were not found in Huacapuy or in Caleta de Quilca. Therefore, it can be confirmed that the results have a significant removal percentage in the removal of anions and in terms of total coliform bacteria and fecal coliforms as a whole.

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ABOUT THE ORGANIZER

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