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EFFICIENCY OF NATURAL COAGULANTS FOR THE REMOVAL OF HEAVY METALS IN WASTEWATER FROM OIL AND GAS DRILLING WELLS

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Abstract: The efficiency of natural coagulants such as *Opuntia ficus* indica and Cassava starch as coagulants in the treatment of water associated with oil and gas production was determined. Laboratory-scale coagulation, flocculation and sedimentation tests were carried out with times of 1 minute (rapid mixing), 20 minutes (slow mixing) and 30 minutes, respectively. The water samples associated with the extraction of oil and gas, with initial turbidity values of 14,500 NTU, the well from which the water sample was taken is called Clarinete-5, Sucre jurisdiction. Various concentrations were evaluated, where parameters such as chemical oxygen demand (COD), turbidity, pH and heavy metals (Mercury, Iron and Lead) were taken into account. The natural coagulant *Opuntia ficus* indica was efficient for the removal of Iron, turbidity, BOD and COD, generating results greater than 87%. While the Cassava starch coagulant was a little less efficient, generating a removal percentage greater than 65% with respect to the parameters studied.
Keywords: Coagulation, flocculation, removal, *Opuntia ficus*-Indica, Cassava Starch.

INTRODUCTION

The oil industry is one of the fastest growing industries and plays a significant role in the economic development of developing countries, as indicated [1]. However, this industry generates wastewater that contains a wide variety of contaminants, including petroleum hydrocarbons, mercaptans, oils, greases and, critically, heavy metals [1] [2].

This industry constitutes one of the most harmful sectors for water resources, analyzing the impact that activities of extractive industries have caused or generate as a potential risk in affecting the environment and especially water. Taking into account the importance of water as a natural resource that sustains life, all efforts must be directed at protecting it and preventing its depletion, but they cannot be aimed at stopping progress, on the contrary, they must find the formulas that allow joint development that not only stimulates the growth of the industry, but also protects water sources.

Current techniques for treating these wastewaters include bioreactor-based technologies such as stirred, membrane, packed-bed, and fluidized reactors (Kuyukina et al., 2020). In addition, other techniques have been used such as membrane technology, photocatalytic degradation, advanced oxidation processes and electrochemical catalysis (Varjani et al., 2019). These techniques are vital, especially when dealing with high concentrations of contaminants, including heavy metals.

Since production generates a high polluting load that causes the deterioration of the quality of life and regulatory non-compliance, which forces the industry to seek measures to minimize environmental impacts. Therefore, this research is necessary to reduce the organic loads in the effluent towards the oxidation lagoon and thus achieve compliance with current regulations [3], using mechanisms in the coagulation processes such as natural coagulants that seek optimization in the removal of heavy metals.

It is essential to highlight that heavy metals present in wastewater, such as cadmium, lead, nickel and vanadium, come from organometallic complexes and represent a significant risk to the environment and human health [1]. Furthermore, wastewater from other sectors, such as rare earth metallurgy, also contains radioactive metals, highlighting the need for effective treatments [2].

The removal of these heavy metals has been shown to be effective using biocatalysts, such as hydrophobic sawdust co-immobilized with Rhodococcus cultures, resulting in a removal of up to 96% of certain heavy metals [2].

Therefore, the behavior of parameters such as pH, turbidity, biological oxygen demand (BOD), chemical oxygen demand (COD) and heavy metals will be studied, seeking to provide an alternative solution to this type of water, using tools low cost such as the implementation of natural coagulants based on cassava starch and *Opuntia ficus* indica.

MATERIALS AND METHOD

In this section, a study was carried out to remove heavy metals present in wastewater from oil and gas drilling wells using natural coagulants based on cassava starch and *Opuntia ficus* indica.

The scope of the correlational-longitudinal research since its purpose is to evaluate the relationships that exist between coagulants made from cassava starch and *Opuntia ficus* indica. The population corresponds to a mass load of residual water, extracted from the mud used in the drilling of hydrocarbon wells, which were supplied by a company in the sector, legally constituted and authorized by the National Hydrocarbon Agency to explore and exploit oil and gas deposits in Colombia, and directs work in different Sedimentary Basins in Colombia. The sample corresponding to the wastewater that comes from the drill cuttings will be evaluated in the following parameters BOD, COD, turbidity, pH, metals such as Hg, Fe and Pb. For the experimental design, it was carried out in three repetitions and three treatments corresponding to wastewater from oil and gas drilling, with different pH; Since this variable was not controlled, three treatments were carried out and the last one was the one that underwent three repetitions. From the data obtained, a study of means and standard deviation was carried out and from these, the analysis of variance was carried out with support in the Dunnett test, since this test is used to make planned comparisons, where each one is compared. of the groups (in this case each jar studied in each of the treatments) with respect to a control group, through this procedure the treatments that present effective conditions with respect to the control are detected. This allowed us to have reference for a total of 40 trials.

In the first stage, the wastewater sample was physicochemically characterized, following the water monitoring protocol and IDEAM guidelines. Samples were taken using simple manual samples in airtight plastic bottles and analyzes of pH, turbidity, BOD, COD and concentration of heavy metals were carried out.

In the second stage, the optimal dose of natural coagulants is calculated. Cassava and *Opuntia ficus* indica were collected and processed, extracting starch and pulverizing the coagulant. A jar test trial was carried out to determine the optimal dosage of the coagulants. Finally, in the third stage, the effectiveness of the coagulant was evaluated. The efficiency of the process was considered through the percentage of turbidity removal and was compared with current environmental regulations.

RESULTS AND DISCUSSIONS

The results obtained during the development of this research are presented below.

PHYSICOCHEMICAL CHARACTERISTICS OF WASTEWATER FROM OIL AND GAS DRILLING WELLS

The results obtained after analyzing the wastewater effluent from oil and gas drilling wells are found in table 2.

PARAMETERS	VALUE OBTAINED	LIMIT VALUES - Res. 0631 OF 2015
pH	11,48	6.00 – 9.00
Turbidity	15825	NR
DBO5	3168,75	200.00 mg O2/L
DQO	5216,14	400.00 mg O2/L
Iron	1971	3,00 mg/L
Copper	1,94	1,00 mg/L
Lead	<LC (0.10)	0,20 mg/L
Chrome	2,66	0,50 mg/L
Nickel	0,51	0,50 mg/L
Arsenic	18,03	0,10 mg/L
Mercury	0,74	0,01 mg/L

Table 2. Characterization results of wastewater from oil and gas drilling wells

Source: Laboratory results, 2022

In general terms, it can be observed that the parameters pH, COD, BOD, Iron, Copper, Chromium, Nickel, Arsenic and Mercury do not comply with the maximum permissible limits according to Colombian legislation Resolution 0631 of 2015, so it is evident that The waters from the extractive activity present an enormous concentration of contaminants as observed in the initial characterization carried out. Thanks to the results obtained, three metals (Lead, Mercury, Iron) were chosen in order to evaluate the percentage of removal of these through the application of a solution of natural coagulants.

DETERMINATION OF THE OPTIMAL DOSE OF NATURAL COAGULANTS BASED ON STARCH EXTRACTED FROM CASSAVA AND OPUNTIA FICUS INDICA

Below are the results obtained at different concentrations, in the treatments and runs carried out, which provided the optimal range of the coagulant dose.

To determine the range of best behavior, three tests and three repetitions were carried out with different concentrations of the coagulant solution; The optimal dose was established taking into account the removal efficiency with respect to turbidity.

Next, the tables will be presented with each of the turbidity values found and in which it was observed that the coagulant obtained greater efficiency.

DETERMINATION OF THE OPTIMAL DOSE FOR THE *OPUNTIA FICUS* INDICA

For this organic coagulant, the range of best behavior fluctuated between 12 and 15 mg/L at 1% for all concentrations studied, however, the best behavior was obtained with 15 mg/L.

In that sense, [4] obtained good results in their water treatment process by applying a 1% solution concentration. The optimal dose for the removal of turbidity and color with respect to the coagulant was 5 mg/L.

Tables 3, 4 and 5 show the behavior of *opuntia ficus indica* during the tests to obtain the optimal dose. It can be seen that for the different concentrations the dose with the best performance was 15 mg/L where the better result against turbidity.

DETERMINATION OF THE OPTIMAL DOSE FOR THE NATURAL COAGULANT BASED ON CASSAVA STARCH

For this organic coagulant, the range of best behavior fluctuated between 7 and 10 mg/L at 1% for all concentrations studied, however, the best behavior was obtained with 8 mg/L.

In this sense [5] in their research Application of the clarifier of natural origin (cassava starch) for the removal of turbidity and color in waters evaluated cassava starch at different concentrations (1%, 2%, 3% 4% 5 % 6%). The optimal concentration found in the set of tests carried out in the jar test was 1%.

Evaluation 1 – Turbidity Values Achieved (NTU)						
DOSE	**5 mg/l**	**30 mg/l**	**55 mg/**	**80 mg/l**	**105 mg/l**	**Control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	110	170	300	650	1.200	3.400
Removal	14.390	14.330	14.200	13.850	13.300	11.100

Table 3.- Evaluation of turbidity with the natural coagulant *opuntia ficus indica* in run 1
Source: Authors, 2022

Evaluation 2 – Achieved Turbidity Values (NTU)						
DOSE	**5 mg/l**	**10 mg/l**	**15 mg/l**	**20 mg/l**	**25 mg/l**	**control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	112	89,6	73,2	99,9	102	3.600
Removal	14.388	14.410,4	14.426,8	14.400,1	14.398	10.900

Table 4.- Turbidity evaluation with the natural coagulant *opuntia ficus indica* in run 2
Source: Authors, 2022

Evaluation 3 – Achieved Turbidity Values (NTU)						
DOSE	**5 mg/l**	**8 mg/l**	**12 mg/l**	**15 mg/l**	**20 mg/l**	**Control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	110	92	85,4	71,6	99	3.600
Removal	14.390	14.408	14.414,6	14.428,4	14.401	10.900

Table 5- Evaluation of turbidity with the natural coagulant *opuntia ficus indica* in run 3
Source: Authors, 2022

Evaluation 1 – Turbidity Values Achieved (NTU)						
DOSE	**5 mg/l**	**30 mg/l**	**55 mg/l**	**80 mg/l**	**105 mg/l**	**control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	135,7	350,6	983,4	1.126	1.500	3.200
Removal	14.364,3	14.149,4	13.516,6	13.374	13.000	11.300

Table 6- Evaluation of turbidity with the natural coagulant based on cassava starch in run 1
Source: Authors, 2022

Evaluation 2 – Achieved Turbidity Values (NTU)						
DOSE	**5 mg/l**	**10 mg/l**	**15 mg/l**	**20 mg/l**	**25 mg/l**	**control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	420	345	600	870,6	1600	3.400
Removal	14.080	14.155	13.900	13.629,4	12.900	11.100

Table 7- Evaluation of turbidity with the natural coagulant based on cassava starch in run 2
Source: Authors, 2022

Evaluation 3 – Achieved Turbidity Values (NTU)						
DOSE	**6 mg/l**	**7 mg/l**	**8 mg/l**	**9 mg/l**	**10 mg/l**	**control**
Initial (NTU)	14.500	14.500	14.500	14.500	14.500	14.500
Final (NTU)	412,2	380,1	311	340	348,7	3.700
Removal	14.087,8	14.119,9	14.189	14.160	14.151,3	10.800

Table 8- Evaluation of turbidity with the natural coagulant based on cassava starch in run 3
Source: Authors, 2022

Tables 6, 7 and 8 show the behavior of cassava starch during the tests to obtain the optimal dose. It can be seen that for the different concentrations, the dose with the best performance was 8 mg/L, where the better result against turbidity.

EVALUATION OF THE EFFECTIVENESS OF COAGULANTS FOR DETERMINING REMOVAL IN TERMS OF PHYSICOCHEMICAL PARAMETERS AND HEAVY METALS

Next, the analysis and discussion of the results of the treatments carried out for wastewater from hydraulic drilling are carried out. First of all, the efficiency of the treatment with the natural coagulants *Opuntia ficus indica* and cassava starch stands out. Efficiencies evaluated for the reduction of COD, BOD, Heavy metals (Pb, Fe, Hg) and pH.

Efficiency of *Opuntia ficus* indica during the treatment of wastewater from hydraulic drilling. It was worked at 1% and with concentrations from 5 mg/L to 105 mg/L. It must be noted that the pH was not taken as a controlled variable, however, it was measured in each of the runs.

Removal of the studied parameters, heavy metals (Pb, Fe, Hg), COD, BOD, and pH from the optimal dose found with the natural coagulant *Opuntia ficus indica.*

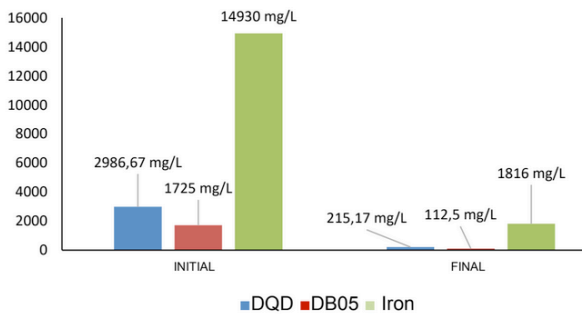

Figure 1. Initial and final evaluation (COD, BOD and IRON) applying *Opuntia ficus* indica

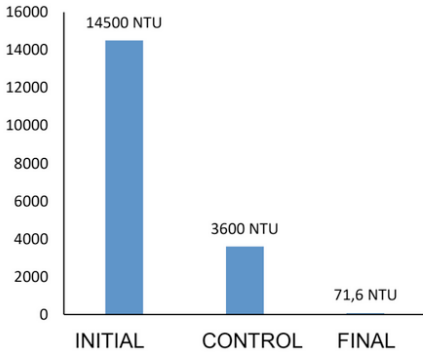

Figure 2. Evaluation of turbidity applying *Opuntia ficus indica.*

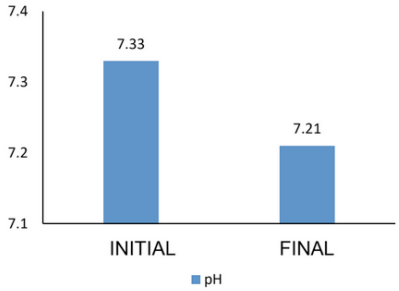

Figure 3. Initial and final evaluation of pH applying *Opuntia ficus indica*

As it can be seen in Figures 1, 2 and 3, the behavior with respect to the removal of the variables studied can be stated that they are directly correlated with turbidity, in this sense parameters such as COD and BOD 5 exceed 90 % removal when applying the optimal dose of *Opuntia ficus* indica coagulant found in the last treatment, in the same way it is shown that the pH decreased from 7.33 to 7.21 and the iron presented 87.9% removal and turbidity more than 95%. In the study [6], antecedents are presented where it is evident that the *Opuntia ficus* indica presents a better behavior for removing turbidity, heavy metals, BOD5 and COD at low pH, managing intervals between 3 - 8.

In the research of [4], when applying, *Opuntia ficus-indica*, at a 1% solution concentration. The optimal dose for the removal of turbidity and color with respect to the coagulant was 5 mg/L. Obtained a removal percentage of 76.85% color and 92% turbidity.

Now, it is necessary to emphasize that, although in the first instance it was chosen to study other metals such as lead (Pb) and mercury (Mg), no trace of these components emerged in the second test sent to the laboratory.

REMOVAL OF THE STUDIED PARAMETERS, HEAVY METALS (Pb, Fe, Hg), COD, BOD, AND pH FROM THE OPTIMAL DOSE FOUND WITH THE NATURAL COAGULANT BASED ON CASSAVA STARCH

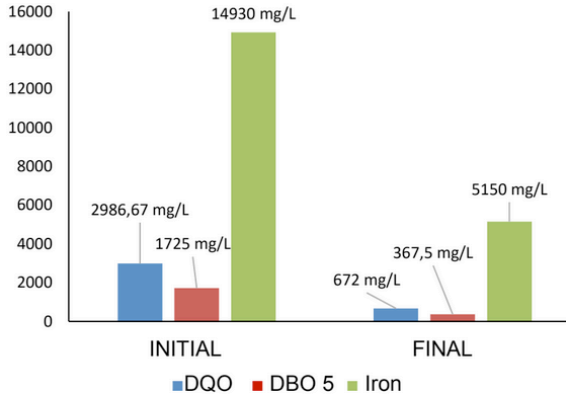

Figure 4. Initial and final evaluation (COD, BOD and IRON) applying cassava starch as a coagulant

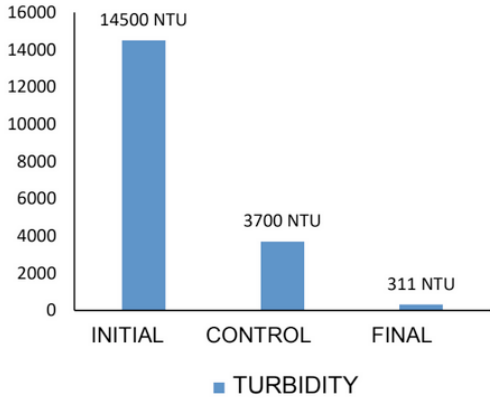

Figure 5. Evaluation of turbidity by applying cassava starch as a coagulant

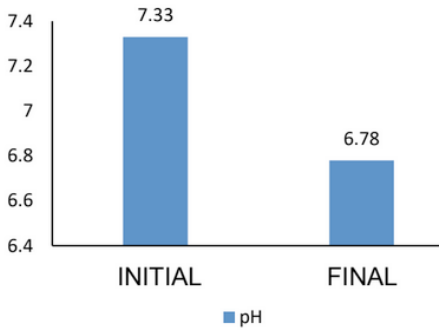

Figure 6. Initial and final evaluation of pH applying cassava starch as a coagulant

As it can be seen in Figures 4, 5 and 6, the behavior with respect to the removal of the variables studied, in this sense parameters such as BOD5, we can observe that the removal percentage was 78.69% and although It is a high value, we observe that it is not enough to comply with the legislation, in the same way they are presented with the other factors such as COD whose removal percentage is 77.47%, iron whose percentage is 65.50%, not However, none of these values comply with the provisions of Resolution 0631 of 2015, which establishes the parameters and maximum permissible limit values in specific discharges to bodies of surface water and public sewage systems and dictates other provisions. In the study of [5] in his research Application of the clarifier of natural origin (cassava starch) for the removal of turbidity and color in waters for human consumption from the Juninguillo–La Mina stream, Moyobamba–San Martín. He evaluated cassava starch at different concentrations (1%, 2%, 3% 4% 5% 6%). The optimal concentration found in the set of tests carried out in the jar test was 1%, that is, 1 mg of starch /L of distilled water (ratio: weight/volume) of cassava starch solution.

Now, it is necessary to emphasize that, although in the first instance it was chosen to study other metals such as lead (Pb) and mercury (Mg), no trace of these components emerged in the second test sent to the laboratory.

COMPARISON OF THE REMOVAL PERCENTAGE IN ACCORDANCE WITH CURRENT ENVIRONMENTAL REGULATIONS

Parameters	Contaminated water	Treated water	Limit values - res. 0631 of 2015
Ph	7,33	7,21	6,00 - 9,00
Turbidity	14.500	71,6	NR
DQO	2.986,67	215,17	400 mg/L
DBO5	1725	112,50	200 mg/L
Mercury	<LC (0,10)	<LC (0,10)	0,01 mg/L
Iron	14.930	1.816	3 mg/L
Lead	<LC (0,119)	<LC (0,119)	0,20 mg/L

Table 9- Comparative table contaminated water water treated with natural coagulant *Opuntia ficus* indica at 1% optimal dose of 15mg/l.

Source: Laboratory Results, 2022

In the previous table you can see the changes that occur in the water associated with hydraulic drilling after having applied *Opuntia ficus* indica at 1%. Which shows a significant reduction in the polluting load.

Thanks to this treatment received, the COD presented a 92.79% removal, the BOD5 presented a 93.47% removal, the Iron 87.83% and the turbidity 99.5%.

It must be noted that the BOD5, COD and pH parameters are within the parameters required by Colombian legislation through Resolution 0631 of 2015 for non-domestic wastewater generated by activities focused on hydrocarbons.

Although The Iron presented 87.83% removal, it does not comply with the requirements of Colombian legislation through Resolution 0631 of 2015 for non-domestic wastewater generated by activities focused on hydrocarbons.

Metal characterization carried out before the application of the coagulant, mercury or lead were not present, this due to the formation environment, they are subject to changes and deformations depending on the deposition environment found at that time, or external environmental factors and internal in the basin which can generate variability in the contaminant load.

From the study carried out it can be inferred that the results obtained in the laboratory regarding the optimal dose of the drug diverge with [6]. Since, our dose for *Opuntia ficus* indica was 15mg/L while in the previous monograph the optimal doses were within the range 4mg/l-10mg/l.

In the same way, it can be deduced that the results obtained in the laboratory with respect to the optimal dose diverge [4]. Since, our dose for *Opuntia ficus* indica is 15mg/L.

In the research it was possible to analyze that there is convergence with [7]. In his research thesis titled "Efficiency of the natural coagulant *Opuntia ficus*"

Likewise having a turbidity removal efficiency of 99%.

Likewise, it was evident that *Opuntia ficus* indica has better performance for removing turbidity, heavy metals, BOD5 and COD in pH between the ranges 3-8, which coincides with the study of [6].

In the last characterization of metals carried out before the application of the coagulant, mercury or lead were not present, this due to the formation environment, they are subject to changes and deformations depending on the deposition environment found at that time, or external and internal environmental factors in the basin which can generate variability in the contaminant load.

Initial Turbidity	Control Jar Turbidity	Turbidity when applying optimal dose of 15mg/l
14500 NTU	3600NTU	71,6NTU

Table 10- Table of turbidity ranges when applying *Opuntia ficus indica*

Source: Authors, 2022

The previous table also shows a dose removal percentage of 71.6 out of 98% compared to the control, which guarantees the effectiveness of *Opuntia ficus* indica in removing turbidity.

Parameters	Contaminated water	Treated water	Limit values - res. 0631 of 2015
Ph	7,33	6,78	6,00 - 9,00
Turbidity	14.500	311	NR
DQO	2.986,67	672	400 mg/L
DBO5	1725	367,50	200 mg/L
Mercury	<LC (0,10)	<LC (0,10)	0,01 mg/L
Iron	14.930	5150	3 mg/L
Lead	<LC (0,119)	<LC (0,119)	0,20 mg/L

Table 11- Comparative table contaminated water water treated with natural coagulant 1% cassava starch where the optimal dose is 8 mg/l.

Source: Authors, 2022

In the table above you can see the changes that occur in the water associated with hydraulic drilling after having applied 1% starch. Which shows a reduction in the pollutant load, but the COD, BOD5 and Iron parameters do not comply with the maximum permissible value required by Colombian legislation through Resolution 0631 of 2015 for non-domestic wastewater generated by the focused activities. in hydrocarbons.

It can be seen that the pH does comply with the maximum permissible value required by Colombian legislation through Resolution 0631 of 2015 for non-domestic wastewater generated by activities focused on hydrocarbons.

The percentage reduction in turbidity was 97.85%, COD 77.5%, BOD5 78.7%, Iron 65%,639.

It must be noted that the BOD5, COD and pH parameters are within the parameters required by Colombian legislation through Resolution 0631 of 2015 for non-domestic wastewater generated by activities focused on hydrocarbons.

Now, at first glance it was possible to analyze a high removal value with respect to turbidity, showing a higher percentage of efficiency compared to [8], where it is stated and studied that the removal percentage in terms of turbidity with cassava starch is 68.5%.

Furthermore, from the study carried out it can be inferred that the results obtained in the laboratory with respect to the optimal dose diverge with [5]. Since our optimal dose for cassava starch is 8 mg/L, this discrepancy may arise because the contaminant load is different.

It was analyzed that there is convergence [5]. In his research, Application of naturally occurring clarifier (cassava starch) for the removal of turbidity and color in waters for human consumption from the Juningui-llo–La Mina stream, Moyobamba–San Martín. He evaluated cassava starch at different concentrations (1%, 2%, 3% 4% 5% 6%). The optimal concentration found in the set of tests carried out in the jar test was 1%, that is, 1 mg of starch /L of distilled water (ratio: weight/volume) of cassava starch solution.

Initial Turbidity	Control Jar Turbidity	Turbidity when applying optimal dose of 8 mg/l
14500 NTU	3700NTU	311NTU

Table 12- Table of turbidity ranges when applying cassava starch

Source: Authors, 2022

A removal percentage of the dose of 311 of 91% with respect to the control is also evident, which guarantees the effectiveness of starch in removing turbidity.

CONCLUSIONS

According to the expected results in the project and the results obtained, we can affirm; that *Opuntia ficus* indica is an effective coagulant that efficiently removes parameters such as COD, BOD5, turbidity and metals such as Iron where removal of up to 90% was obtained.

At the time of characterizing the water, it was observed that it did not comply with the permissible values of resolution 0631 except for pH. However, after finding the optimal dose with the *Opuntia ficus* indica, it was achieved that it met the permissible values in COD, BOD5 parameters.

It can be concluded that in comparison of both coagulants, the most effective was *Opuntia ficus* indica, having a turbidity removal percentage of 99.5%, COD presented 92.79% removal, and BOD5 with 93.47%. removal and Iron 87.83%.

The pH range in which cassava starch acted best was pH 7.12, showing that at low pH its performance and effectiveness is much more noticeable, being able to eliminate a BOD5 removal percentage of 78.69, COD whose percentage of removal was 77.47 and iron with a percentage of 65.50, however, none of these values comply with the provisions of resolution 0631.

In the last characterization of metals carried out before the application of the coagulant, mercury or lead were not present, this due to the formation environment, they are subject to changes and deformations depending on the deposition environment found at that time, or external and internal environmental factors in the basin which can generate variability in the contaminant load.

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