



LIFE Project Number
LIFE12 ENV/ES/000265

**DELIVERABLE D.A.1.1: REPORT OF THE PRE-INDUSTRIAL
ASSESSMENTS RESULTS IN INDUSTRIAL WASTE WATER (TEXTILE
AND CERAMICS SECTORS).**

**ADNATUR: Demonstration of natural coagulant use advantages in
physical & chemical treatments in industry and urban waste water.**



EXECUTIVE SUMMARY

D.A.1.1 summarizes the first studies and validation carried with wastewater from three end-users at laboratory level. It has been compared the results between the usual procedure and with different doses of the natural coagulants.

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1. INTRODUCTION

ADNATUR main goal is the validation, assessment and industrial demonstration of a new innovative and environmentally friendly technology. This technology is based on products derived from natural extracts, for its use in the primary treatment of wastewaters, at urban and industrial level.

The main advantages of this new technology are technical and environmental improvements, deriving in energy and resources save and the no production of hazardous chemical waste during the physical-chemical treatment of industrial or urban wastewater. In order to demonstrate these advantages two wastewater treatment industrial-scale prototype plants will be designed, assembled, and put into operation in different real end-users facilities. The proposed technique will be demonstrated at industrial level in two Spanish companies from textile and ceramic sector and at urban wastewater level.

This specific action is focussed on natural based coagulant validation procedure. In order to do so, the coagulant efficiency at lab scale will be tested in real wastewater samples coming from textile and ceramics sectors. Thus, several Jar Tests using increasing dosage of natural coagulants will be carried out. The efficiency of natural coagulants will be compared with the efficiency of currently used inorganic coagulants. Raw materials used for developing natural coagulants comes from the same substrate, extracted from acacia, and is chemically modified in order to improve its coagulant efficiency. This modified coagulant will be used in the industrial demonstration phase.

In this specific action the main advantages that will be demonstrated are:

- Reduction of corrosive tendency and/or fouling of water, increasing the lifetime of the facilities in contact with treated water,
- Increase of the security of workers due to the substitution of hazardous products by eco-friendly products.
- Minimize the use of chemicals such as coagulants and flocculants, avoiding neutralizing agents in the physical-chemical treatment, controlling salt supplies.
- Improvement of biological processes and sludge dehydration.
- Reduction of costs derived from hazardous wastes management.

2. EXPERIMENTAL PROCEDURE

In order to validate the new technology based in natural coagulant a coagulation-flocculation test at lab scale will be carried. Standard procedures are called Jar Test. In this test a device with the name of flocculator is used. This has five or six points of homogenization, which allows, at a certain speed, simultaneous stirring of the liquid contained in several beakers. It is very important that during the test, water sample has approximately the same temperature than in the real treatment plant.

First, raw wastewater (500 ml) is stirred in different beakers. Next step is to add the coagulant with agitation between 100 and 150 revolutions per minute (rpm), because in this step a quick agitation is needed. In order to have a proper action of coagulant, the agitation is maintained between 3 and 10 minutes, depending of the retention time in the real installation. Then, a pH corrector is added if it is necessary. The dosage of polyelectrolyte or flocculant is the final step and, in this case, the speed of the stirrer is reduced to 60 rpm in order to achieve maturation and growth flocculate. The time needed for this step is between 2 and 10 minutes, after this, the stirrer is switched off.

When agitation is stopped, formed flocs are deposited. The time of sedimentation is between 5 and 30 minutes. After this, a sample of the clarified water is analysed to determine diverse physicochemical parameters, such as chemical oxygen demand (COD), suspended solids (SS) or turbidity, with the objective of quantify the quality of the treatment. Taking into account that several samples can be treated at the same time, the influence of different kind of coagulants and flocculants can be tested. Several parameters can be modified, as time of reaction or velocity of the stirring and, after that, the flocs sedimentation time and the quality of the clarified obtained must be experimentally determined.

Once all the process has been carried out and the optimum dosages have been obtained, the Jar Test is repeated with the same sample and with the same combinations of coagulants, but adding the total amount of products in one step only. When the dosage is gradual, the reaction time is longer than with one-step dosage and results could be not realistic. With this final test, the reproducibility of the treatment and the dosages of coagulants have been assessed.

3. RESULTS AND DISCUSSION

3.1. CHARACTERIZATION OF WATER SAMPLES.

Water in the industry is involved in many different ways and in most cases an essential key in the production system. Some of the water is extracted from own supplies (wells) or from the drinking water network. Part of the water is lost during the process, and the other part is generated in the form of wastewater. Following, a summary of the kinds of resultant waters is detailed:

- Industrial wastewater, as the result of the different manufacturing steps.
- Sanitary wastewaters comparable to urban that are the consequence of the population of workers involved in the processes.
- Cooling circuit wastewater that is used in the heat exchange between equipment (presses, motors, cogeneration...) and refrigerant (water).
- Process cut and polished of ceramic tile wastewater. In these processes the water acts as refrigerant of the grinding wheels and cutting saws and as a vehicle for the particles eroded tiles.

The characteristics of each kind of waters are very different, for this reason the problems derived from the treatment of wastewater are very diverse and specific. As has been said in the introduction, during the development of this new technology, two kinds of water coming from industrial wastewater (ceramic and textile sector) will be treated. The characterization of different waters will be shown in the next points.

3.1.1. CERAMIC SECTOR.

In the ceramic industry many wet processes are given, which implies high consumption of water. Companies included in this sector, carried out manufactured ceramic tiles finishes. This means chamfering and/or polishes the surface to achieve different finishes. For this purpose, highly abrasive grinding wheels made with substances that are consolidated by inorganic salts and resins are used.

In this process, water is used for cooling both the grinders and cutting and polishing saws and for dragging the eroded particles from the ceramic pieces. The main characteristics of this kind of water are:



- High salinity caused by continuous supply of salts from the abrasive used in the polishing process and a minor fraction from inorganic coagulants used in their treatment. However, this water does not have problems of organic matter, heavy metals and boron.
- High conductivity values cause corrosion and fouling problems on facilities and pipes and, for instance, greatly reducing the average life of them.

In this type of water is essential establishing an appropriate system of purges in the circuit in order to keep the salt content between appropriate values.

The samples of the ceramic sector used for the study of the new technology based on natural coagulants are from two different processes. The first sample is from ceramic grinding. A complete characterization of that sample is shown in Table 1.

PARAMETER	Method	Units	Result
pH	ELM/001	u pH	8,2
Conductivity 20 °C	ELM/004	μS/cm	2210
Chlorides	VOL/005	mg/l	319
Turbidity	NEF/001	NTU	800
Chemical Oxygen Demand (COD)	EAM/006	mgO ₂ /l	32
Suspended solids	GRA/001	mg/l	2939
Sulphates	EAM/011	mg/l	460
Calcium	VOL/002	mg/l CaCO ₃	1225

Table 1. Ceramic grinding wastewater characterization.

The second sample is from ceramic glazes. A complete characterization of that sample is shown in Table 2.

PARAMETER	Method	Units	Result
pH	ELM/001	u pH	7,0
Conductivity 20 °C	ELM/004	μS/cm	2520
Chlorides	VOL/005	mg/l	390
Turbidity	NEF/001	NTU	800
Chemical Oxygen Demand (COD)	EAM/006	mgO ₂ /l	1905
Suspended solids	GRA/001	mg/l	10000
Sulphates	EAM/011	mg/l	150
Calcium	VOL/002	mg/l CaCO ₃	830

Table 2. Ceramic glazes wastewater characterization.

In the Tables 1 and 2, the parameters analysed in water samples used in this study are indicated, as well as, the internal laboratory method.

3.1.2. TEXTIL SECTOR.

The textile industry consumes large quantities of water and produces large volumes of wastewater from different steps such as tanning, dyeing and finishing processes. For this reason, wastewater treatment represents an important environmental issue. Consequently, correct management is necessary for water reuse in the textile industries. Wastewater from textile industry is often rich in color, containing residues of reactive dyes and chemicals, high COD and BOD concentration, high values of conductivity as well as much more hard-degradation materials.

The sample collected for the study of the new technology base in natural coagulant is from tanning industry. The tanning industry is of a considerable pollution load in terms of both organic and toxic parameters.

A complete characterization of the sample from textile sector, more specifically from tanning is shown in Table 3.

PARAMETER	Method	Units	Result
pH	ELM/001	u pH	7,3
Conductivity 20 °C	ELM/004	μS/cm	5080
Chlorides	VOL/005	mg/l	532
Turbidity	NEF/001	NTU	> 1000
Chemical oxygen demand (COD)	EAM/006	mgO ₂ /l	3145
Total nitrogen	EAM/010	mg/l	150
Suspended solids	GRA/001	mg/l	2515

Table 3. Textile wastewater characterization.

In the Table 3, the parameters analysed in water samples used in this study are indicated, as well as, the internal laboratory method.

3.2. PHYSICAL-CHEMICAL TREATMENTS.

After the characterization of the different water samples that will be used in the study of ADNATUR technology, an efficiency comparison between inorganic coagulants and natural based coagulants has been carried out. The main objectives to be achieved in the current laboratory scale study are:

- Assess efficiency of ADNATUR technology in front of inorganic coagulants in wastewater.

- Verify that natural base coagulants reduce corrosive tendency and/or fouling of water. Lifetime of the facilities in contact with treated water will be extended because of non-toxic and environmentally friendly character of the products used in the eco-innovative technology. Moreover, security of workers will be also improved.
- Alkalinity of the medium is not consumed during water treatment because of the chemical properties of the technology. So, the use of neutralizing agents like HCl, NaOH or HNO₃, currently required, will be reduced or even avoided in physical-chemical treatment of wastewater. Thus, a decrease in the use of chemicals (in some cases reaching 30% reduction) during the process will be experimentally demonstrated.
- Regarding its strong cationic character and its optimal coagulant efficiency in a wide range of pH, proposed technology provides a rapid flocculation and decantation, even at lower chemical dosage values. For this reason, coagulant and flocculant dosage will be reduced. In the case of coagulant the reduction of product is of 35% and in the case of flocculant, dosage reduction is close to 10% approximately.
- Control the salts supply to the treated water to not damage the current production system. Furthermore, with non-incorporation of metallic elements as Iron or Aluminum, the next steps like biological processes or sludge dehydration will be clearly favoured.
- All the advantages mentioned also produce direct costs reduction, fairly estimated in a 40%. This due to the limitation of used containers and management of generated residues, reduction on the chemicals dosage and the present environmentally friendly character of the new products.

As has been mentioned previously, in order to compare the different coagulants at laboratory scale, the Jar Test method will be used.

3.2.1. CERAMIC SECTOR.

In order to develop the study of ceramic grinding wastewater, a sample of inlet water was collected. In the current procedure two coagulants are compared: an inorganic coagulant, in this case poly aluminum chloride (PAC), and ADNATUR technology, the coagulant named ADNATUR K50.



ADNATUR K50 is a natural coagulant based on tannin with a strong cationic character. The raw material is extracted from the bark of the Black Acacia (*Acacia mearnsii*), which has a strong coagulant action. The tannin extract is chemically modified in order to improve its coagulant efficiency.

In Table 4 and 5, the most remarkable results of the Jar-Test carried out in SERVYECO laboratory have been shown. With the coagulant ADNATUR K50 the use of any neutralizing agent is not required. In contrast, with the use of poly aluminium chloride some sodium hydroxide is needed in order to have a working pH value of 8. Additionally, ECOPOL AS 575 (Anionic Solid Flocculant) was used in the second part of the treatment.

Products/ppm	1	2
Poly Aluminum Chloride	70	-
ADNATUR K50	-	50
NaOH	10	0
Stirring speed (rpm)	100	100
Stirring time (sec)	20"	20"
Sedimentation (*)	L	M
Turbidity (**)	8	8

Table 4. Coagulation test result.

*S: Sedimentation → R (decanted in less than 4 seconds) M (decanted in less than 10 seconds.)

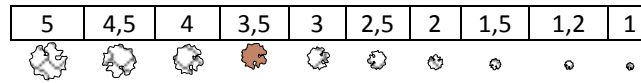
L (decanted in more than 10 seconds) F (floats)

**T: Turbidity → Scale (1-10) of the clarified water after 20", 10 being the best clarified.

Products/ppm	1	2
Poly Aluminum Chloride	70	-
ADNATUR K50	-	50
NaOH	10	0
ECOPOL AS575	2	1
Stirring speed (rpm)	60	60
Stirring time (sec)	3'	3'
Floc diameter*	4	4,5
Turbidity**	10	10
Resistance***	40	85
Sedimentation****	M	R

Table 5. Flocculation test result.

*D: Floc diameter (under laboratory conditions, the floc is usually lower than the real scale)



**T: Turbidity → Scale (1-10) of the clarified water after 20'', 10 being the best clarified.

***R: Resistance: Size ratio after subjecting the floc to mechanical stress: $\% = \left(\frac{D_F}{D_I} \right) \times 100$

****S: Sedimentation → R (decanted in less than 4 sec.) M (decanted in less than 10 sec.)

L (decanted in more than 10 sec.) F (floats)

It is observed that the best results are achieved by using ADNATUR technology based on natural coagulants. Clarified water with good value of turbidity and proper separability is achieved with a reduction in the coagulant dosage of 30%. With respect to the formed floc after the use of the tested coagulants, the flocs formed with ADNATUR K50 exhibit a higher degree of dehydration and higher resistance.

Then, the analytical results obtained from the resulting clarified are detailed:

0.- Inlet water.

1.- Clarified water after 70 ppm of Poly Aluminum Chloride and 2 ppm of **ECOPOL AS575**.

2.- Clarified water after 50 ppm of **ADNATUR K50** and 1 ppm de **ECOPOL AS575**.

PARAMETER	Method	Units	0	1	2
pH	ELM/001	u pH	8,2	8,0	8,2
Conductivity 20 °C	ELM/004	µS/cm	2210	2450	2210
Chlorides	VOL/005	mg/l	319	420	301
Turbidity	NEF/001	NTU	800	15,5	10,3
Chemical Oxygen Demand (COD)	EAM/006	mgO ₂ /l	32	32	30
Suspended solids	GRA/001	mg/l	2939	42	17
Sulphates	EAM/011	mg/l	460	460	460
Calcium	VOL/002	mg/l CaCO ₃	1225	789	680

Table 6. Clarified water characterization.

REMOVAL RATE (%)	1	2
Conductivity 20 °C	-10,8	0
Chlorides	-31,7	5,64
Turbidity	98,1	98,7
Chemical Oxygen Demand (COD)	0	6,25
Suspended solids	98,6	99,4
Sulphates	0	0
Calcium	35,6	44,5

Table 7. Removal rate of the different parameters.

From the results obtained it can be concluded that the coagulant ADNATUR K50, besides not increasing conductivity of the sample, reduces it. It should be underlined that conductivity is one of the most problematic parameters in ceramic sector wastewater. Furthermore, it also reduces the concentration of COD, suspended solids and calcium, as well as the turbidity of the clarified further than the inorganic coagulant poly aluminum chloride. Finally highlight that the inorganic coagulant increases chlorides concentration while natural coagulant reduces it.

For ceramic glazes wastewater, a sample of inlet water was also collected. In the study, as with ceramic grinding wastewater, two coagulants are compared, an inorganic coagulant, poly aluminum chloride (PAC), and ADNATUR technology, the coagulant ADNATUR K50.

In Table 8 and 9, the most representative results of the Jar-Test carried out in SERVYECO laboratory have been shown. With the coagulant ADNATUR K50 the use of any neutralizing agent is not required. In contrast, with the use of poly aluminum chloride some sodium hydroxide is needed in order to have a working pH value of 8. Additionally, ECOPOL AS 575 (Anionic Solid Flocculant) was used in the second part of the treatment.

Products/ppm	1	2
Poly Aluminum Chloride	800	-
ADNATUR K50	-	500
NaOH	60	0
Stirring speed (rpm)	100	100
Stirring time (sec)	20''	20''
Sedimentation (*)	L	M
Turbidity (**)	7	8

Table 8. Coagulation test result.

* S: Sedimentation → R (decanted in less than 4 seconds) M (decanted in less than 10 seconds.) L (decanted in more than 10 seconds) F (floats)











**T: Turbidity → Scale (1-10) of the clarified water after 20', 10 being the best clarified.



Products/ppm	1	2
Poly Aluminum Chloride	800	-
ADNATUR K50	-	500
NaOH	60	0
ECOPOL AS575	2	1
Stirring speed (rpm)	60	60
Stirring time (sec)	3'	3'
Floc diameter*	4	4,5
Turbidity**	9	10
Resistance***	40	85
Sedimentation****	M	R

Table 9. Flocculation test result.

*D: Floc diameter (under laboratory conditions, the floc is usually lower than the real scale)

5	4,5	4	3,5	3	2,5	2	1,5	1,2	1
									

**T: Turbidity → Scale (1-10) of the clarified water after 20'', 10 being the best clarified.

***R: Resistance: Size ratio after subjecting the floc to mechanical stress: $\% = \left(\frac{D_F}{D_I} \right) \times 100$

****S: Sedimentation → R (decanted in less than 4 sec.) M (decanted in less than 10 sec.)

L (decanted in more than 10 sec.) F (floats)

It is observed that the best results are achieved by using ADNATUR technology based on natural coagulants. Clarified water with lower turbidity value compared to the use of inorganic coagulant is achieved and, additionally, proper decantation with a reduction of the coagulant dosage, reaching 38%. With respect to the formed floc after the use of the tested coagulants, the flocs formed with ADNATUR K50 exhibit a higher degree of dehydration and higher resistance.

Then the analytical results obtained from the resulting clarified are detailed:

0.- Inlet water.

1.- Clarified water after 800 ppm of Poly Aluminum Chloride and 2 ppm of **ECOPOL AS575**.

2.- Clarified water after 500 ppm of **ADNATUR K50** and 1 ppm de **ECOPOL AS575**.

PARAMETER	METHOD	UNITS	0	1	2
pH	ELM/001	u pH	7,0	8,0	7,0
Conductivity 20 °C	ELM/004	µS/cm	2520	3010	2600
Chlorides	VOL/005	mg/l	390	530	461
Turbidity	NEF/001	NTU	800	210	15
Chemical Oxygen Demand (COD)	EAM/006	mgO ₂ /l	1905	1520	1500
Suspended solids	GRA/001	mg/l	10000	88	75
Sulphates	EAM/011	mg/l	150	170	32
Calcium	VOL/002	mg/l CaCO ₃	830	800	800

Table 10. Clarified water characterization.

REMOVAL RATE (%)	1	2
Conductivity 20 °C	-19,4	-3,17
Chlorides	-35,9	-19,2
Turbidity	73,8	98,1
Chemical Oxygen Demand (COD)	20,2	21,3
Suspended solids	99,1	99,3
Sulphates	-13,3	78,7
Calcium	3,61	3,61

Table 11. Removal rate of the different parameters.

From the results obtained it can be determined that coagulant ADNATUR K50 does not substantially increase conductivity compared to poly aluminum chloride. Furthermore, it also reduces the concentration of COD, suspended solids and calcium, as well as the turbidity of the clarified further than the inorganic coagulant poly aluminum chloride. Finally highlight that the inorganic coagulant increases sulphates concentration while natural coagulant reduces it.

3.2.2. TEXTILE SECTOR.

In order to carry out the study of textile wastewater, a sample of inlet water was collected. In the study two coagulants are compared, an inorganic coagulant, in this case ferric chloride, and ADNATUR technology, the coagulant ADNATUR K70.

In Table 12 and 13, the most representative results of the Jar-Test study carried out in SERVYECO laboratory will be shown. When adding the coagulant ADNATUR K70, the use of any neutralizing agent is not required. In contrast, with the use of ferric chloride, calcium hydroxide is needed in order to have a working pH value of 8. Additionally, ECOPOL AS 575 (Anionic Solid Flocculant) was used in the second part of the treatment.

Products/ppm	1	2
Ferric chloride	700	-
ADNATUR K70	-	450
Ca(OH) ₂	240	0
Stirring speed (rpm)	100	100
Stirring time (sec)	20"	20"
Sedimentation (*)	L	M
Turbidity (**)	8	8

Table 12. Coagulation test result.

* S: Sedimentation → R (decanted in less than 4 seconds) M (decanted in less than 10 seconds.)

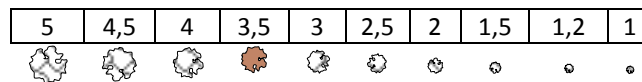
L (decanted in more than 10 seconds) F (floats)

**T: Turbidity → Scale (1-10) of the clarified water after 20', 10 being the best clarified.

Products/ppm	1	2
Ferric chloride	700	-
ADNATUR K70	-	450
Ca(OH) ₂	240	0
ECOPOL AS575	4	3,5
Stirring speed (rpm)	100	100
Stirring time (sec)	3'	3'
Floc diameter*	4	4,5
Turbidity**	9	10
Resistance***	70	85
Sedimentation****	M	R

Table 13. Flocculation test result.

*D: Floc diameter (under laboratory conditions, the floc is usually lower than the real scale)



**T: Turbidity → Scale (1-10) of the clarified water after 20", 10 being the best clarified.

***R: Resistance: Size ratio after subjecting the floc to mechanical stress: $\% = \left(\frac{D_F}{D_I}\right) \times 100$

**** S: Sedimentation → R (decanted in less than 4 sec.) M (decanted in less than 10 sec.)



L (decanted in more than 10 sec.)

F (floats)

Best results are achieved by using ADNATUR technology based on natural coagulants. Clarified water with lower value of turbidity and proper separability is achieved, even reducing the coagulant dosage up to 36%. With respect to formed flocs after the use of the tested coagulants, with ADNATUR K70 they exhibit a higher degree of dehydration and increased resistance. The dosage of flocculant is also reduced approximately a 10% with the use of ADNATUR K70.

Below, the analytical results obtained from the resulting clarified are detailed:

0.- Inlet water.

1.- Clarified water after 700 ppm of **Ferric chloride** and 4 ppm of **ECOPOL AS575**.

2.- Clarified water after 450 ppm of **ADNATUR K70** and 3,5 ppm de **ECOPOL AS575**.

PARAMETER	METHOD	UNITS	0	1	2
pH	ELM/001	u pH	7,3	8,0	7,3
Conductivity 20 °C	ELM/004	µS/cm	5080	5400	5085
Chemical Oxygen Demand (COD)	EAM/006	mgO ₂ /l	3145	760	745
Chlorides	VOL/005	mg/l	532	815	656
Total nitrogen	EAM/010	mg/l	150	122	102
Suspended solids	GRA/001	mg/l	> 1000	15,9	13,1
Turbidity	NEF/001	NTU	2515	33	28

Table 14. Clarified water characterization.

REMOVAL RATE (%)	1	2
Conductivity 20 °C	-6,30	-0,10
Chemical Oxygen Demand (COD)	75,8	76,3
Chlorides	-53,2	-23,3
Total nitrogen	18,7	32,0
Suspended solids	>98,4	>98,7
Turbidity	98,7	98,9

Table 15. Removal rate of the different parameters.

From the results obtained it can be determined that the coagulant ADNATUR K70 does not increase conductivity compared to ferric chloride. Furthermore, it also reduces COD, suspended solids and total nitrogen concentration, as well as the turbidity of the clarified water, even further than the inorganic coagulant, ferric chloride.

4. CONCLUSIONS

After the improvement of the tannin extract from *Acacia mearnsii*, new products with strong cationic character and very high coagulant efficiency, called ADNATUR K50 and K70, have been developed and validated at laboratory scale.

ADNATUR technology is compared with the most commonly used coagulants in wastewater treatment, inorganic coagulants. For this purpose, several Jar Tests have been carried out in the laboratory of SERVYECO. Current treated samples come from ceramic and textile sector.

The conclusions obtained after the tests are described bellow:

- ADNATUR technology is more efficient than current inorganic coagulants as poly aluminum chloride and ferric chloride. With this technology up to 30% of coagulant dosage has been reduced at laboratory scale.
- The strong cationic character of ADNATUR technology, gives the formed flocs a higher degree of dehydration and increased resistance. For this reason, a reduction in the use of flocculant is also possible, up to 10%.
- Additionally, the dosage reduction or even elimination of other chemicals, such as neutralizing agents, has been also demonstrated. Natural coagulants do not consume the alkalinity of the medium. Its coagulant efficiency is always optimal because it does not suffer hydrolysis in solution and have a proper coagulant action in a wide range of pH (between 4.5 and 9). For this reason, in the most of cases a pH adjust is not required when dosing ADNATUR products. However, with inorganic coagulants the use of some neutralizing agent such as sodium hydroxide or hydrochloric acid is mostly necessary.
- As a consequence of the reduction or elimination of pH regulators the sample conductivity was not increased. Thus, corrosive tendency and/or fouling of treated water are reduced and lifetime of the equipment in contact with treated water will be extended.
- Substitution of hazard chemicals as ferric chloride or hydrochloric acid for eco-friendly products improves security of worker, reduces waste to be treated and improves next steps in water treatment, including biological processes or sludge dehydration. Furthermore, the sludge produced with ADNATUR technology can be used, in some cases, for agricultural purposes.