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Optimizing Absorbent Bentonite and Evaluation of Contaminants Removal from Petrochemical Industries Wastewater

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Abstract: *The aim of this research was to show the better use of internal resources and increased capability of utilizing and optimizing minerals. Also, in this research we tried to present a new way of petrochemical wastewater treatment. In this sample of bentonite, after the production process in microwave through digestion method with Nano Precision, a long chain of detergents was processed and optimized with an organic material (dimethyl octadecyl ammonium chloride). In the beginning of tests, a statistical method was drawn by MINITAB software and Taguchi method. Sampling was made in different times and testing started by a standard method. Among measuring factors, 5 factors of turbidity, TSS, TDS, pH, and COD were evaluated and their changes were investigated in different times based on the amount and type of materials. The effluent COD and BOD are very important. The petrochemical wastewater was measured and it was90ppm(COD) and 3 ppm(BOD). Also, the highestlevel of turbidity for material A (modified bentonite) was 1.2 NTU with 10mg/L of sludge. Considering that the country needed a better wastewater treatment through environment protection, modification of bentonite's structure, and nano application process in this industry along with various available methods, we aimed to take a step towards the development of the country.*

Keywords: *Bentonite, wastewater treatment, design of Taguchi method tests, turbidity, COD, TDS, TSS*

1. INTRODUCTION

Inattention towards wastewater treatment operations can lead to contamination of water and soil resources and, consequently, infectious and chronic diseases. Physical, chemical, and biological wastewater treatment methods are of special importance. Moreover, given the limitation of water sources, when wastewater is treated appropriately and is compatible with environmental standards, the treated wastewater can be reused in different fields such as agriculture (1). Protection of valuable sources, like bentonite soil, and their use in the treatment of industrial wastewater are from effective solutions for improving the level of environmental cleanliness (2).

The purpose of this research was, first of all, the optimization of bentonite soil and then the use of this material to remove different pollutants from industrial (petrochemical) wastewater. Petrochemical wastewater is very dangerous and contains various pollutants.

Bentonite is a mineral from the category of clays or pseudo-clays generated from inflated minerals. In general, it contains montmorillonite and a little *beidellite*. For the same reason, bentonite is a clay species which contains 85%-90% of montmorillonite mineral. The term "bentonite" was introduced for the first time in 1898 by Knight as clay, and then extracted from Benton Shale. This termis said to have come from the name of a region called Montmorillon in the south of France, containing large sources of bentonite (3, 4). On the other side, bentonite is also referred to as clay mainly composed of smectite minerals (regardless of its origin) with the following chemical formula(5):

$(Na,Ca)_{0.33}$ $(Al,Mg)_2Si_4O_{10}$ $(OH)_2$. nH_2O.

The different types of bentonite are each named after the respective dominant element, such as potassium (K), sodium (Na), calcium (Ca), and aluminium (Al). Experts debate a number of nomenclatorial problems with the classification of bentonite clays. Bentonite usually forms from weathering of volcanic ash, most often in the presence of water. However, the term bentonite, as well as similar clay called tonstein, has been used to describe clay beds of uncertain origin. For industrial purposes, two main classes of bentonite exist: sodium and calcium bentonite (3-5). Bleaching property and the quantity of water absorbed by bentonite depend on the type of mineral(s) of smectite group. The swellrate and bleaching properties of bentonites is normally not desirable; as a result, it is necessary to make some modifications so that its properties increase. Bentonite is made of alumina and silica flakes with weak bonds which can be separated in aqueous media to 0.003 micrometers thick and 0.1 micrometer length. Easy separation property and negative electric charge cause their wide spread in water. Bentonite contains interchangeable cations Ca^{2+}, Na^{2+} or $Mg^{2+}$and has a higher ion exchange capacity than other minerals except zeolite. This property impacts its commercial characteristics and its classification takes place on the same basis, such as sodium bentonite with a very high ability to swell and calcium bentonite with a poor ability to swell. Another major difference between these two types of minerals is that the type containing sodium is stable at a temperature of 400°C. On the other hand, calcium bentonite reactions with sodium carbonate so that its swelling property is increased (6, 7).

Due to properties such as softening and swelling, a relatively good ability to be mixed with water, plasticity, adhesiveness, absorbency and ..., bentonite is widely used in various areas like the production of drilling mud, casting sand preparation, preventing water leak in dams and irrigation channels, preparation of transparent liquids such as fruit juices, acting as water clarifier and smoother of liquids like paraffin, pelletizing of minerals including iron ore, pelletizing of animal feed, acting as carrier agent in paints and other spray products, preparation of animal and plant toxins, filler in industries like papermaking, production of cleaners and detergents, production of ceramics, bleaching, and treatment of oils (8).

The main research question was whether modified bentonite can be reducing parameters of COD, TDS, and TSS effluent to be effective?

For this purpose, 1 sample of raw bentonite,2 types of modified bentonite and 2 types of activated bentonite and also 1 type of nano-bentonite was used. The limitation of study was included: Lack of financial resources and low reproducibility (3 times).

Although this is a relatively new topic, but several studies have been conducted on the benefits and uses of bentonite in wastewater treatment. Studies related to the research subject-matter included:

- Natural zeolites is effective in water treatment by Margeta et al., (2013);
- Evaluation of natural zeolites in the purification and separation of gases by Ackley et al., (2003);
- Investigation of the possibility of removing copper, nickel and lead from aqueous solutions using bentonite by Futalan et al., (2012);
- Using bentonite in chemical treatment of industrial wastewater by Sahu and Chaudhari (2013);
- Removal of emerging contaminants from water and wastewater by adsorption process with materials such as bentonite by Grassi et al., (2012);
- Evaluation of the application of bentonite to remove heavy metals from wastewater by Al-Jlil (2010);
- Evaluation of bentonite soil modification using sulfate and anionic phosphate for the removal of heavy metals by Olu-Owolabi and Unuabonah (2010);
- Feasibility of using sodium bentonite in removing heavy metals by Triantafyllou et al., (1999);
- Evaluation of the treatment of wastewater contaminated with cobalt using Saudi activated bentonite by Al-Shahriani (2013). (9 – 17).

2. MATERIALS AND METHODS

This was an applied research as for outputs and a cross-sectional one in terms of application time. Basic information was gathered firstly through investigating theoretical fundamentals in previous literature. Then we began to identify issues related to wastewater in petrochemical industries by holding brainstorming sessions with the participation of engineers of refineries and petrochemical sites. To do tests, the wastewater of Khorasan Petrochemicals Company and the bentonite available in Kerman were used.

For a more precise identification of forming minerals, the bentonite sample in the laboratory of Golgohar-e Sirjan Company was analyzed through X-ray diffraction (XRD) method. Due to their structural formulation, these materials absorb OAT and reduce its quantity; therefore, this quantity can be diminished by modifying and/or optimizing the bentonite.

Next, five types of bentonites (raw bentonite D; modified bentonite A and B; calcium bentonite E, sodium bentonite F and German nano bentonite C) were supplied and tested.

2.1. Bentonite Optimization Method

The purpose of optimization was raising the level of active contact surface and better efficiency catalytic properties cations sodium, calcium and potassium exchange.Organoclay method was used in order to optimize the tested bentonite (18).Organoclay is an organically modified phyllosilicate, derived from a naturally occurring clay mineral(19)(20). By exchanging the original interlayer cations for organocations (typically quaternary alkyl ammonium ions) an organophilic surface is generated, consisting of covalently linked organic moieties. The lamellar structure remains analogous to the parent phyllosilicate. Organoclay manufacturing process was carried out without the application of heat and acid activation. For this purpose, 2 g of the raw bentonite sample was prepared which was gradually added to 500cc water; then it was stirred by a mechanical shaker for 1 hour at a speed of 160 rpm. 2.4 g of dimethyl octadecyl ammonium chloride was added. This time the blending speed was increased to 120 rpm, and then it was continued to blend for 2 hours. All these steps were conducted at room temperature, and drying was done after 2 hours. It should be noted that the bentonite powder shall not be filteredandbe dried with the same water inside it (21)(22). A vacuum oven at a temperature of 30°C can be used to accelerate the drying process.

Table 1 and Table 2 illustrate the analysis of raw and modified bentonite samples.

Table1. *Analysis of raw bentonite Dsample*

TiO_2	MgO	CaO	K_2O	Na_2O	Fe_2O_3	Al_2O_3	SiO_2
0.8 %	5.22 %	1.04 %	0.35 %	3.09 %	5.96 %	14.77 %	60.34%

Table2. *Analysis of bentonite samples tested*

Compound name	Loi	TiO_2	MgO	CaO	K_2O	Na_2O	Fe_2O_3	Al_2O_3	SiO_2
Modified bentonite A and B	7.70%	0	5.22%	1.04%	35%	3.09%	5.96%	14.77%	60.38%
Calcium bentonite B	7.87%	0	4.81%	25%	29%	4.64%	6.27%	12.74%	60.68%
Sodium bentonite F	0	269	1.86	1.68	2.831	9.3	1.67	7.631	68.72
German Nano-bentonite C	15.45%	62%	3.29%	1.97%	86%	98%	5.62%	19.60%	50.95%

Table3. *Analysis of the wastewater entering the treatment plant*

pH	**TEMP (°c)**	**Change in pH**	**Turbidity (NTU)**	**EC (ms)**	**TDS (ppm)**	**COD (ppm)**
7.05	22.5	3	16	1.96	2300	3400

Taguchi method was used in the design of experiments. The main effective factors included materials and the rate of changes in pH studied at 5 levels.

3. CONCLUSION AND DISCUSSION

During the sampling in several stages and at different times and, then, the measuring of all desired parameters, the quality of the wastewater entering the treatment plant of the petrochemical company was determined as follows:

According to Taguchi method, the wastewater sample tested was measured with each of the given bentonites. The results are as shown in Table 4.

Table4. *Results of wastewater analysis after its contact with selected bentonites*

Sample	pH	Change in pH	Turbidity (NTU)	EC (ms)	TDS (ppm)	Settled sludge (g/lit)
A	2.50	2.42	1.26	3.95	2.96	10
A	5.00	4.30	2.30	2.84	3.12	25
A	7.50	7.00	4.00	2.50	3.00	25
A	10.00	8. 6	4. 5	3.00	1.10	15
A	12.50	10.00	20.00	2.88	15.00	15
B	2.50	3.00	20.50	4.00	2.96	60
B	5.00	5.80	27.40	2.85	2.13	50
B	7.50	7.40	40.00	3.00	2.00	50
B	10.00	9.60	76.00	2.00	1.98	30
B	12.50	12.00	86.00	1.90	3.20	50
C	2.50	2.60	36.00	3.95	2.96	90
C	5.00	5.60	40.05	2.84	2.13	120
C	7.50	7.00	40.02	1.46	1.10	200
C	10.00	10.50	50.00	1.22	0.95	180
C	12.50	12.00	76.90	1.23	0.92	160
D	2.50	2.42	8.50	1.00	2.96	10
D	5.00	4.50	12.00	0.90	2.13	15
D	7.50	7.00	19.00	0.98	1.10	15
D	10.00	9.00	31.00	0.95	0.95	10
D	12.50	11.00	79.00	0.90	0.92	20
E	2.50	3. 3	5.00	1.32	0.95	20
E	5.00	6.00	6.20	1.38	0.92	25
E	7.50	7.50	7.00	1.23	0.96	25
E	10.00	9.00	8.00	1.80	0.95	30
E	12.50	10.60	9.30	2.00	0.92	25
F	2.50	2.90	10.90	1.50	0.80	15
F	5.00	4.00	13.09	1.43	0.94	20
F	7.50	6.30	21.30	1.56	0.98	25
F	10.00	8.40	32.90	1.65	0.95	20
F	12.50	10.50	40.60	1.43	0.95	25

According to the results obtained from 6 samples of bentonite, the sample A had the highest turbidity level compared with other samples of bentonite. Similarly, COD was measured and it was 90ppm and also, BOD was measured and it was 3 ppm. These results indicate that the modified bentonite had affected the wastewater sample (Table 5).

Table5. *Results of using selected bentonite (type A) on the wastewater sample from petrochemical industries*

Parameter	Pre-treatment	Post-treatment
BOD	1000	3
COD	5000	90
TDS	5000	40
TSS	1000	180
TIFF	18000	6
EC	640	2
Hardness	700	300
Sulfate	5000	1880
Chloride	300	120
Inlet flow	-	300-2000

Figure 1 shows the main effects of the sample for S/N ratios.

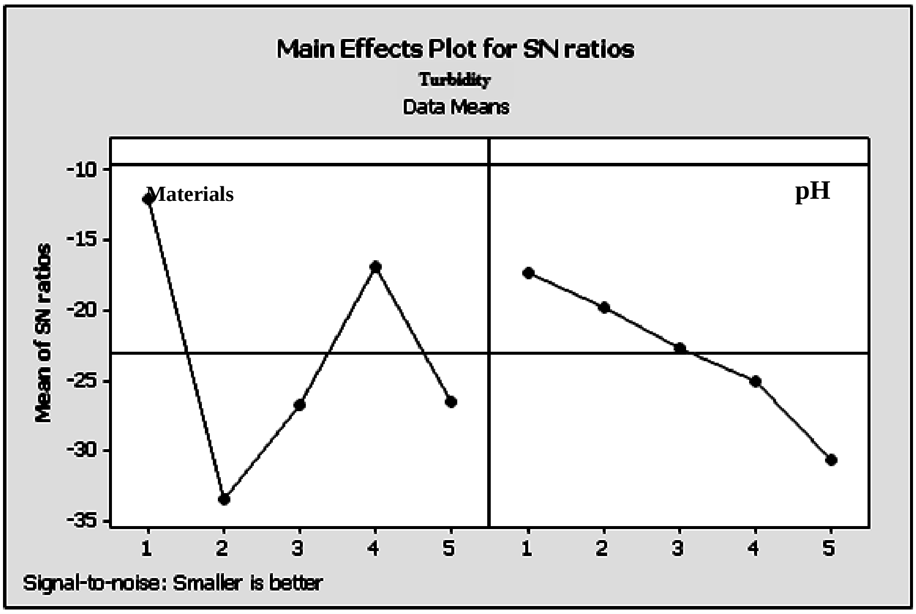

Fig1. *Results of the main data related to petrochemical wastewater*

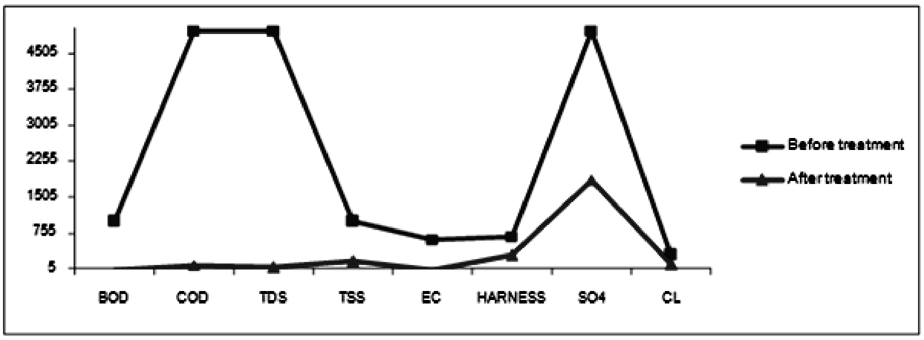

Fig2. *Comparison of parameters tested with bentonite (A) pre- and post-treatment*

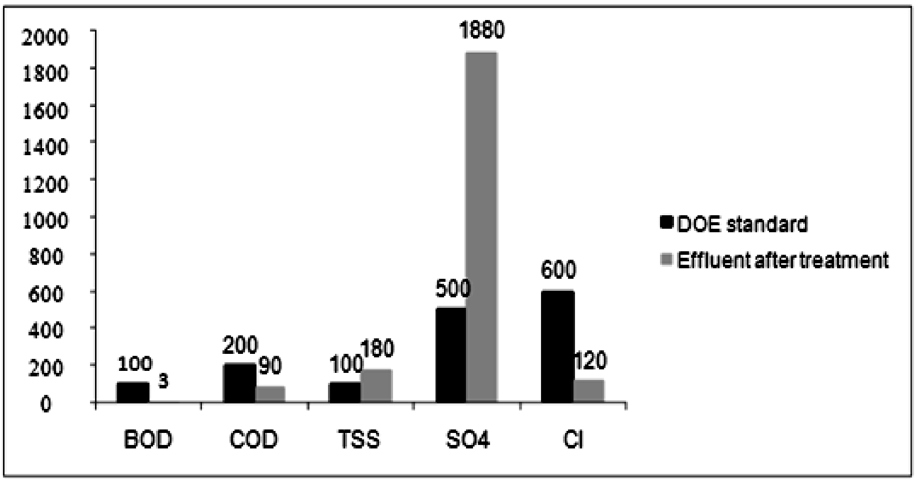

Fig3.*Comparison of the results of wastewater treatment with bentonite (A) and national standards to use wastewater for irrigation of landscape (DOE, 2012)*

The diagram shown above illustrates the effect of materials and pH on turbidity. It should be noted that absolute numbers (SN) are taken into consideration in this diagram so that the smallest data indicates the best result. The effect of material on turbidity is investigated at the left side, while the effect of pH on turbidity is examined at the right side (23)(24)(25). Firstly, material number 1, bentonite A, has the highest effect on turbidity, and the material number 2, German nano bentonite, has the lowest effect. In this section, the impact of materials is considered regardless of the pH effect. On the left, pH diagram indicates that the more acidic is the solution, the higher its effect will be (i.e.

type 1 with an acidity of 2.5), and the more alkaline it is (i.e. type 2 with an alkaline pH of 12.5), the lower its effect will be. The effect of pH on turbidity is linear. The more the acidity, the greater the turbidity and the alkalinity level in wastewater indicates that its conditions are far from ideal. However, this is not true for the effect of materials (26)(27).

This diagram shows that the materials have no interaction (i.e. decrease and/or increase of one factor has no effect on the other). Materials, pH, and their influence line are the same.

It should be noted that the smallest values in diagrams above represent the best values and the absolute valuesshall be taken into consideration. The effect of the change in the materials and the pH is the same (equal). Any line on the pH diagram indicates a pH value and on the materials diagram indicates a material. According to Table 6, which shows the effect of bentonite materials and pH, the turbidity obtained is desirable for all samples. Also, the volume of the settled sludge has decreased. The optimal condition was for material a (modified bentonite) and with a turbidity of 1.2 and a sludge volume of 10 mg/L. The turbidity level is completely consistent with national and international standards (28)(29)(30).

Table6. *Final data on the petrochemical wastewater*

Materials	pH	Change in pH	Turbidity (NTU)	EC (ms)	TDS (ppm)	Settled sludge (g/lit)	SNRA1	MEAN1
A	2.5	2.42	1.26	3.95	2.96	10	-2.00741	1.26
A	5	4.3	2.3	2.84	3.12	25	-7.23456	2.3
A	7.5	7	4	2.5	3	25	-12.0412	4
A	10	8. 6	4. 5	3	1 .098	15	-13.0643	4.5
A	12.5	10	20	2.88	0.9	15	-26.0206	20
C	2.5	2.6	36	3.95	2.96	90	-31.1261	36
C	5	5.6	40.05	2.84	2.13	120	-32.0521	40.05
C	7.5	7	40.02	1.46	1.095	200	-32.0455	40.02
C	10	10.5	50	1.22	0.95	180	-33.9794	50
C	12.5	12	76.9	1.23	0.92	160	-37.7185	76.9
D	2.5	2.42	8.5	0.9	2.96	10	-18.5884	8.5
D	5	4.5	12	0.9	2.13	15	-21.5836	12
D	7.5	7	19	0.98	1.095	15	-25.5751	19
D	10	9	31	0.95	0.95	10	-29.8272	31
D	12.5	11	79	0.9	0.92	20	-37.9525	79
E	2.5	3.3	5	1.32	0.95	10	-13.9794	5
E	5	6	6.2	1.38	0.92	15	-15.8478	6.2
E	7.5	7.5	7	1.23	0.96	15	-16.902	7
E	10	9	8	1.8	0.95	10	-18.0618	8
E	12.5	10.6	9.3	2	0.92	20	-19.3697	9.3
F	2.5	2.9	10.9	1.5	0.8	15	-20.7485	10.9
F	5	4	13.09	1.43	0.94	20	-22.3388	13.09
F	7.5	6.3	21.3	1.56	0.98	25	-26.5676	21.3
F	10	8.4	32.9	1.65	0.95	20	-30.3439	32.9
F	12.5	10.5	40.6	1.43	0.95	25	-32.1705	40.6

Table 7depicts the effect of each of the bentonite's material and the pH. For example, material A has an 88% impact on the wastewater.

Table7. *Effect of materials and pH on the petrochemical wastewater*

Materials	**Materials effect**	**pH effect**	**pH**
A	88%	12%	2.5
C	64%	36%	5
D	73%	27%	7.5
E	80%	20%	10
F	70%	30%	12.5
TOTAL	21.31	13.35	

The obtained results show that all tested parameters are significantly reduced and improved by the Type abentonite post-treatment. Compared with the existing standards on the use of treated wastewater to be used for irrigation of landscape and agriculture, however, sulfate and TSS are still higher than approved despite their decrease post-treatment (than their initial condition), and the use of complementary methods is therefore required(31)(32).

Results indicate that sample 1 (bentonite A) has been the best efficiency. Optimum condition was in pH= 2.5 (Acidic). Thus, modified bentonite (sample A), have been better results than German bentonite (sample C). In addition to lower cost of this bentonite is an advantage.

The innovative aspect of this research included using of new modification method for bentonite and also simultaneous use of several types of bentonite to petrochemical wastewater treatment and compares the results.

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