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Precipitation of Ortophosphate in a Wastewater Treatment Plant

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Abstract:	Commercial concentrations of different individually applied salts are generally used to precipitate some contaminants in wastewater. In this research, we studied a complementary treatment to the lagoon system of a wastewater treatment plant by adding a combination of trivalent metal salts to chemically precipitate phosphate. Laboratory tests were carried out to determine the most efficient treatment for the removal of phosphate in wastewater, by analyzing orthophosphate using the ascorbic acid method. Three trivalent metal salts: iron (III) sulfate, aluminum (III) sulfate, and iron (III) chloride, were combined with each other in three different concentrations, obtaining an experimental design with 27 treatments replicated 3 times. An analysis of variance (ANOVA) with Tukey test was applied, identifying the treatment that obtained the highest percentage of orthophosphate removal with 99.04%. Finally, the selected treatment was implemented for its field evaluation for three weeks, obtaining a removal average of 93.13%.
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Precipitation of Ortophosphate in a Wastewater Treatment Plant

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Abstract

Commercial concentrations of different individually applied salts are generally used to precipitate some contaminants in wastewater. In this research, we studied a complementary treatment to the lagoon system of a wastewater treatment plant by adding a combination of trivalent metal salts to chemically precipitate phosphate. Laboratory tests were carried out to determine the most efficient treatment for the removal of phosphate in wastewater, by analyzing orthophosphate using the ascorbic acid method. Three trivalent metal salts: iron (III) sulfate, aluminum (III) sulfate, and iron (III) chloride, were combined with each other in three different concentrations, obtaining an experimental design with 27 treatments replicated 3 times. An analysis of variance (ANOVA) with Tukey test was applied, identifying the treatment that obtained the highest percentage of orthophosphate removal with 99.04%. Finally, the selected treatment was implemented for its field evaluation for three weeks, obtaining a removal average of 93.13%.

Keywords Wastewater, Chemical treatment, Precipitation, Metal salts.

1. Introduction

Water pollution is an increasing global concern and one of the most important environmental problems. It causes damages in the aquatic ecosystems since it modifies the biological, physical, and chemical characteristics of water. Additionally, it can cause severe public health threats (Moe and Rheingans 2006; Zhang et al. 2011). Human activities are the main origin of this pollution, which is produced in multiple ways. An important one is the industrial discharge, which releases to water bodies different compounds, such as dyes, pesticides, heavy metals, etc. (Aniagor and Menkiti 2018; de Castro Schwab and Cammarota 2018; Demcak et al. 2017; UN-Water 2017). Some of these industrial effluents are no able to be biodegraded by biological treatment organisms in treatment plants (Almeida et al. 2004).

Wastewater treatment can be carried out through physical, chemical or biological processes (Castro Bohórquez and Hernández Perilla 2017; Safoniuk 2004).

High phosphate level promotes algae growth and limits the activity of microorganisms responsible for the reduction of organic load, affecting biological treatments and generating eutrophication processes (Kim et al. 2013). Orthophosphate, the nutrient with a highest level of eutrophication, causes problems with water quality and maintenance of oxidation ponds where treatments are given (Lundehøj et al. 2019;

Montes 2016). In order to reduce nutrients and avoid eutrophication phenomena chemical precipitation processes were implemented. Metallic salts and coagulants of organic origin or polymers were used for this purpose (Ma et al. 2018; Nassef 2012).

Phosphorous from wastewater may be present as orthophosphates (PO_4^{-3}), condensed inorganic phosphates (polyphosphates and metaphosphates) and organic phosphates (R. Huang et al. 2018; Saavedra Sanchez 2016). Phosphorus removal is commonly performed by chemical precipitation and / or by applying biological methods (EBPR process, enhanced biological phosphorus removal) (Alejandro H. Caravelli, Edgardo M. Contreras 2008). The main phosphate removal processes are differentiated according to the precipitant point of application; the main aspects are: prior precipitation (precipitants added before pre-treatment), simultaneous precipitation (precipitants added in one or more stages of treatment) and subsequent precipitation (precipitants added after biological treatment) (Maroneze et al. 2014).

High operational costs are limitations in primary treatments, which include precipitation, adsorption or ion exchange (Teles et al. 2020). There are different studies that demonstrate the effectiveness of iron and aluminum salts in wastewater treatment; these salts reduce organic matter, suspended solids and even heavy metals significantly (Kazi et al. 2013; Lopes et al. 2011; Salama et al. 2015; Salminen et al. 2015). Many works demonstrate the effectiveness of aluminum sulfate, ferric sulfate and ferric chloride regarding orthophosphate removal (Jiang and Graham 2003; Lopes et al. 2011; Menezes et al. 2010; S. M. Park et al. 2015). One of the main problems for using these salts individually is the high production of sludge (Isarain-Chávez et al. 2014; Wysocka and Sokołowska 2016), which could be controlled by reducing the amount of milligrams per liter to be used.

On average, high-income countries treat about 70 % of local and industrial wastewater generated. This average drops to 38 % in upper-middle-income countries and to 28 % in lower-middle-income countries. In low-income countries only 8% receive any treatment. These estimates support the commonly cited approximation that more than 80 % of wastewater is discharged without treatment worldwide (WWAP 2017).

The 2008 Constitution of the Republic of Ecuador establishes the regulations and public policies to protect and good practice of water rights that all companies and wastewater treatment plants must comply regarding Article 14, which recognizes the right of the population to live in a healthy and ecologically balanced environment that guarantees sustainability and well-being (Congreso Nacional de Ecuador 2015). However, in this part of the American continent only 10 to 25 % of the plants perform a treatment before returning the water to the natural environment, furthermore, the majority with problems of efficiency and operability (Sato et al. 2013).

Ecuador's local governments mostly treat wastewater through oxidation ponds for example in Manta, with a system consisting in pre-treatment, three anaerobic lagoons, three facultative lagoons and four maturation. This system is no longer suitable because of the high pollutants load produced by a constant growing population. There is also a saturation of solids and ionic species present in aerobic lagoons responsible for the removal of organic load (Velasco T. et al. 2019).

2. Materials and methods

Prior to the execution of the experiment and taking into account the trivalence of the metals of the salts to be evaluated in the experiment, a selection of chemical precipitants was implemented, considering their affinity to the orthophosphate ion (Devlin et al. 2019; Li et al. 2019; Omwene et al. 2018). The reactive grade (Fisher brand) salts were provided by Biomed Instruments and were $Fe_2(SO_4)_3 \cdot 6H_2O$ ferric (III) sulfate hexahydrate, $Al_2(SO_4)_3 \cdot 18H_2O$ aluminium (III) sulfate octadecahydrate and $FeCl_3 \cdot 6H_2O$ ferric (III) chloride hexahydrate.

Wastewater samples were collected in Manta´s wastewater treatment plant (Aguas de Manta Public Company, Ecuador). Particularly, samples were obtained from the reactor where the water from the pre-treatment state is homogenized, prior to the effluent leaving the anaerobic treatment lagoons. At this collection point, reducing the orthophosphate levels does not affect biological processes (Kosse et al. 2019).

The experimental research was developed in two stages; the first one on a laboratory scale, by mounting tests in three replicates for each treatment, where statistical analysis was implemented for the analysis of the, The second stage was developed in the field by implementing a pilot on-site test.

a. Experimental design

The experimental design used in this research work consisted of the using of three factors that correspond to trivalent metal salts. Each of these factors was composed by 3 levels of concentrations expressed in mg/L, as shown in Table 1 (Hinkelmann 2012).

[Table 1 near here]

A total of 27 treatments were established, these treatments resulted from the combination of the levels of each factor, as shown in Table 2, which were replicated 3 times.

[Table 2 near here]

b. Jar test

The jar test methodology was followed using an ESICO model 1926 kit, 1000 mL of sample was taken for each treatment with a class A VAT volumetric balloon to each combined salt. This was added on an ADAM model PW254 balance, where the agitation process was carried out in two parts: initially at 250 rpm for 5 minutes and then at 40 rpm for 20 minutes (Bouyer et al. 2005). After the agitation process, it was left to rest for an additional 20 minutes and then 25 mL of each treatment was taken with their replicates for the orthophosphate analysis.

c. Orthophosphate analysis

The protocol described in technique 4500-P E., contained in the Standard Methods for the Examination of Water and Wastewater - 22nd Edition, was followed. This method is based on the reaction of ammonium molybdate ($(NH_4)_2MoO_4$), antimony and potassium tartrate ($C_8H_{10}K_2O_{15}Sb_2$) in an acid medium. The presence of orthophosphates forms heteropolyacids, specifically phosphomolybdic acid ($H_3PMo_{12}O_{40}$), this reduces to molybdenum which has an intense blue color due to the action of ascorbic acid ($C_6H_8O_6$) (“APHA, AWWA, WEF. ‘Standard Methods Exam. water wastewater.’” 2012).

A calibration curve with concentrations from 5 to 1000 mg/L (11 points, one every 100 mg/L) of orthophosphate was implemented from a orthophosphate stock (1000 mg/L). This stock was prepared with anhydrous monopotassium phosphate. Each sample, both before and after treatment, was analyzed according to the protocol. Concentration was calculated from the calibration curve using the linear regression equation (1).

$$y = mx + b \quad (1)$$

Where y = is the absorbance signal of the sample at 880 nm; m is the slope of the line; b is the y-intercept and x is the calculated orthophosphate concentration.

d. Tabulation and statistic

Microsoft Excel 2016 and InfoStat software were used for data analysis and analysis of variance (ANOVA), respectively. Additionally, in order to determine the significant differences between the different treatments, the Tukey test was implemented. Finally a Pearson correlation coefficient was performed to establish whether there is a dependency between the different treatment results (McHugh 2011).

e. Pilot test

Once the most efficient treatment was selected on the laboratory, this was implemented on a pilot scale on a larger wastewater sample.

For the implementation of this test, a homemade prototype reactor with a capacity of 15 L was built. The prototype was designed taking into account the following parts: a hose for the entrance of the water to be treated, two valves for the exit of the precipitate, a valve for the outlet of treated water and a motor coupled with a fan that rotates constantly at 240 rpm (Figure 1).

[Figure 1 near here]

The reactor was filled weekly during 3 consecutive Tuesdays at 3:00 p.m. At this time, water was pumped from the homogenization reactor to the anaerobic treatment lagoon. Having a single stirring speed, it was decided to carry out stirring for 10 minutes and then wait an extra 60 minutes in order to take the treated water sample for its respective analysis.

3. Results

Table 3 shows the values obtained after running the calibration curve with a wavelength of maximum absorption at 880 nm. Those values that did not fit the curve were discarded using statistical cleaning, to have a curve with at least 5 points, whose coefficient of correlation is greater than 0.987 (Moosavi and Ghassabian 2018).

[Table 3 near here]

The initial concentration of orthophosphate in the effluent to be treated was calculated with an average value of 801.67 mg PO_4^{-3}/L (Table 4). It is known that, high levels of orthophosphates can be caused by the discharge of industrial effluents to the system (Baawain et al. 2015; Henze et al. 2015; Mostafa and Peters 2012; RAMALHO 1983).

[Table 4 near here]

Each of the treatments proposed in the experimental design were repeated three times, reaching an average removal rate of more than 50% in almost all treatments. As an exception the a1.b1.c2 treatment with 49.92% removal capacity. On the other hand, the treatment with the highest percentage of removal was a3.b2.c1, reaching a 99.04% of removal capacity (Figure 2). Removal percentages above 95% are unusual at different laboratory scales (Ahmad et al. 2020; Hamdan and Mara 2013; Han et al. 2015; Onyango et al. 2007; T. Park et al. 2017; Ragheb 2013; Song et al. 2011).

[Figure 2 near here]

According to the average values of each treatment, the divergence effect was found through an analysis of variance with the summation of squares type I (Table 5).

[Table 5 near here]

This ANOVA was added to a Tukey test where 16 differentiation ranges were established (A, B, C , D, E, F, G, H, I, J, K, L, M, N, O, P) between treatments in all the repetitions (Table 6).

[Table 6 near here]

On average, the pilot test using the a3.b2.c1 treatment managed to reduce 93.13% of orthophosphates. This percentage is 5.91% lower than the one resulted from the laboratory test. Similar studies confirm the elimination of phosphorus in its various forms does not depend solely of the resource used for its removal, there are also other factors, such as the interaction with other substances, atmospheric conditions and even the variation of the hydraulic load (Bali and Gueddari 2019; Bekele et al. 2019; X. Huang et al. 2020; Ilyas and Masih 2018).

4. Conclusions

The different combinations of salts tested on a laboratory scale showed statistically different behavior since Tukey's test showed 16 different ranges. Only ranges A, B and C were well-defined.

On the other hand, more than half of the treatments were above 80% removal. This high removal percentage confirms the efficiency of using combined salts when removing orthophosphates.

Finally, the in situ pilot test showed the possibility of optimizing resources by using a combination of salts (a3.b2.c1) with a total concentration of 25 mg / L (4 mg/L of Ferric Sulfate $Fe_2(SO_4)_3$, 8 mg/L of Aluminum Sulfate $Al_2(SO_4)_3$ and 13 mg/L of Ferric Chloride $FeCl_3$) below the individual theoretical optimal concentration of 40 mg/L considered for each salt.

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Declaration of interest statement

The authors declare that there are no conflicts of interest.

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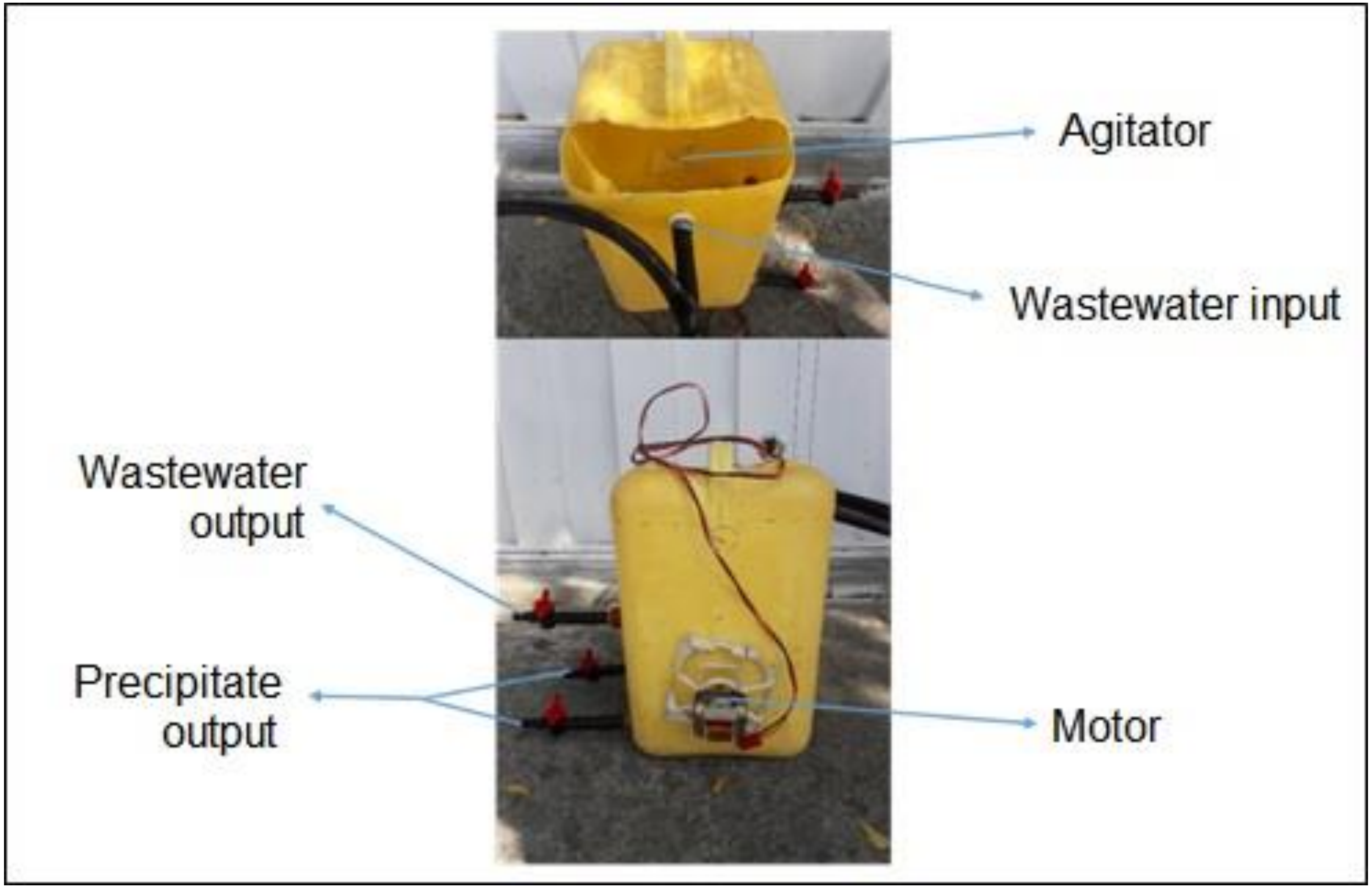

Figure 1. Prototype reactor structure

Figure 2. Orthophosphate removal percentages

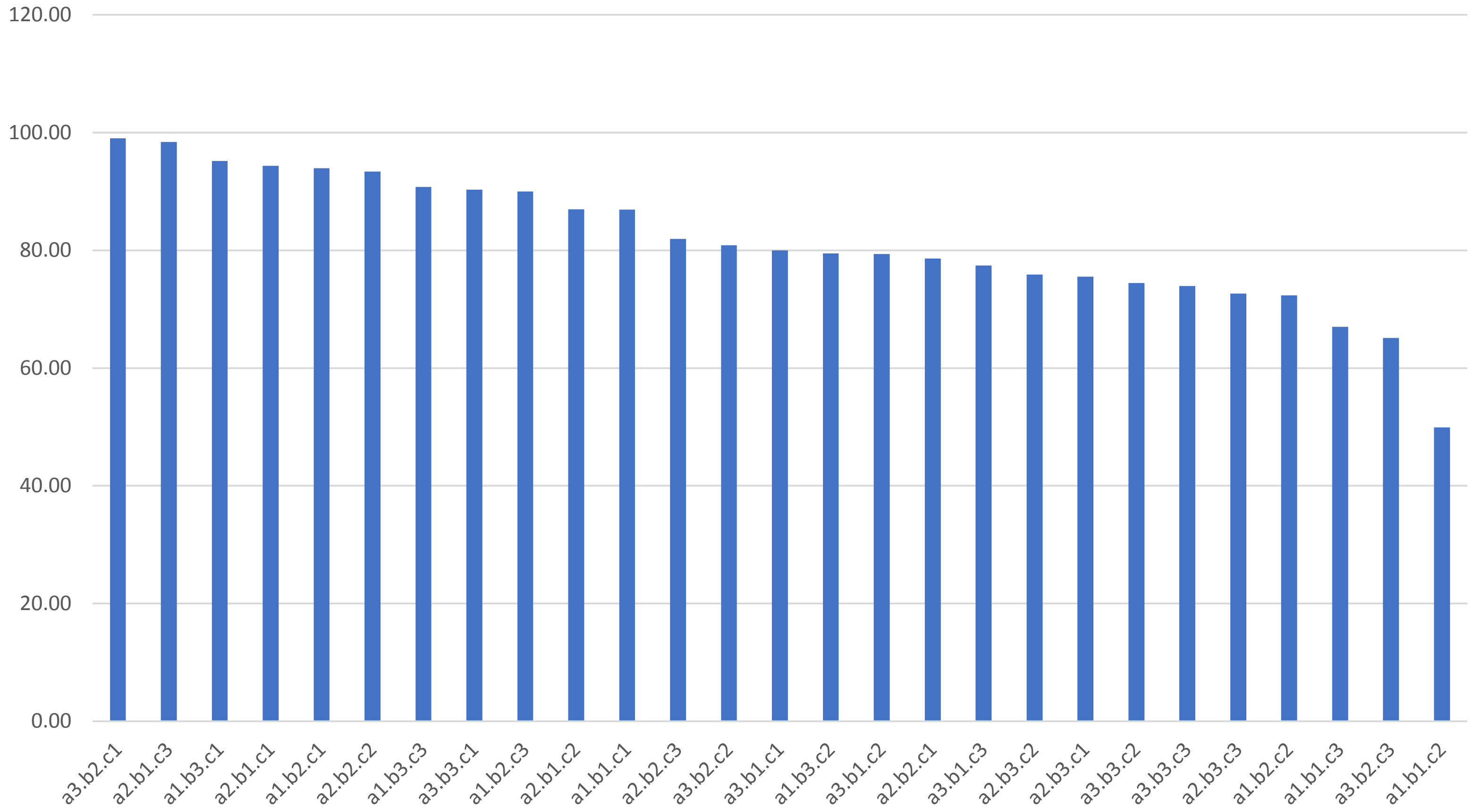

Figure 2. Orthophosphate removal percentages

Table 1. Levels of experimental design

Factors	***A***		***B***		***C***	
	$Fe_2\ (SO_4)_3$		$Al_2\ (SO_4)_3$		$Fe\ Cl_3$	
Levels	a_1	13 mg/L	b_1	13 mg/L	c_1	13 mg/L
	a_2	8 mg/L	b_2	8 mg/L	c_2	8 mg/L
	a_3	4 mg/L	b_3	4 mg/L	c_3	4 mg/L

Table 2. Treatments of experimental design

a_1. b_1. c_1	a_2. b_1. c_1	a_3. b_1. c_1
a_1. b_1. c_2	a_2. b_1. c_2	a_3. b_1. c_2
a_1. b_1. c_3	a_2. b_1. c_3	a_3. b_1. c_3
a_1. b_2. c_1	a_2. b_2. c_1	a_3. b_2. c_1
a_1. b_2. c_2	a_2. b_2. c_2	a_3. b_2. c_2
a_1. b_2. c_3	a_2. b_2. c_3	a_3. b_2. c_3
a_1. b_3. c_1	a_2. b_3. c_1	a_3. b_3. c_1
a_1. b_3. c_2	a_2. b_3. c_2	a_3. b_3. c_2
a_1. b_3. c_3	a_2. b_3. c_3	a_3. b_3. c_3

Table 3. Calibration curve for calculating orthophosphate concentration

Patrón (mg/L)	**ABS**
5	0.973
100	0.998
300	1.03
400	1.065
500	1.1
700	1.15
900	1.195
1000	1.237
R^2	0.9928
m	3785.8546
b	-3651.7070

Table 4. Effluent characterization

	Repetition	**mg PO_4^{-3}/L**	
		Individual	**Average**
Sample without treatment	1	789.50	801,67
	2	812.00	
	3	803.50	

Table 5. Analysis of variance (ANOVA – SS type I)

V.S.	SS	df	MS	F	p-value
Model	646284.50	26	24857.10	1163.66	<0.0001
TREATMENT	646284.50	26	24857.10	1163.66	<0.0001
Error	1153.50 54	21.36			
Total	647438.00	80			

Table 6. Tukey test (Alfa=0.05 MSD=14.70759)

TREATMENT	Mean	n	Ranges					
a3.b2.c1	7.67	3	A					
a2.b1.c3	12.83	3	A					
a1.b3.c1	38.67	3		B				
a2.b1.c1	45.17	3		B				
a1.b2.c1	48.33	3		B				
a2.b2.c2	53.17	3		B				
a1.b3.c3	73.83	3			C			
a1.b2.c3	80.00	3			C			
a2.b1.c2	104.33	3				D		
a1.b1.c1	105.00	3				D		
a2.b2.c3	144.83	3					E	
a3.b2.c2	153.50	3					E	F

a3.b1.c1	160.33	3	F	G									
a1.b3.c2	164.33	3	F	G									
a3.b1.c2	165.50	3	F	G									
a2.b2.c1	171.67	3		G	H								
a3.b1.c3	180.83	3			H	I							
a2.b3.c2	193.17	3				I	J						
a2.b3.c1	196.17	3					J	K					
a3.b3.c1	204.33	3					J	K					
a3.b3.c2	204.67	3					J	K	L				
a3.b3.c3	209.00	3						K	L	M			
a2.b3.c3	219.17	3							L	M			
a1.b2.c2	221.83	3								M			
a1.b1.c3	264.33	3									N		
a3.b2.c3	279.83	3										O	
a1.b1.c2	401.50	3											P