

Chemical Treatment for Removal of Heavy Metals from Industrial Wastewater

H. I. Abdel-Shafy

Water Research & Pollution Control Department, National Research Centre, Dokki (Affiliation ID: 60014618), Cairo, Egypt.

THE OBJECTIVE of the present study is treatment of industrial wastewater that is artificially contaminated with a combination of soluble salts of Ni, Cu and Mn in the concentration of 10 mg/l each. The treatment is focused on employing chemical coagulants and coagulant aids for the removal and co-precipitation of heavy metals. Variable doses of the chemical coagulants at different pH were examined. The physical and chemical characteristics and the level of Cu, Ni and Mn in the industrial wastewater are given. When Sodium hydroxide was employed at pH 9.5, removal rate reached 97.5, 96.0 and 90.0% for Cu, Ni and Mn, respectively. On the other hand, the use of 50 mg/l ferric chloride in combination with sodium hydroxide at different pH was studied. At pH 12.0, all the studied metals were removed at the rate of 98%. When 70 mg/l alum was employed in combination with NaOH at different pH, results indicated that 100% of Cu, 100% of Ni and 84% of Mn were removed at pH 12.0. Further study was carried out using lime at different doses. At pH 11.0, the removal rate reached over 100% for Cu and Ni each and 93% for Mn. Limestone (CaCO_3) at different doses was investigated. Using 1.0 g/l limestone raised the pH from 2.0 to 5.85 and the removal of Cu, Ni and Mn reached 100, 90.2 and 75.1% respectively. By increasing the CaCO_3 dose to 3.0 g/l, more than 100% of Cu and Ni each was achieved while Mn was removed at 90.6%. It was noticed that increasing the limestone dose up to 5.0 g/l did not increase the pH more than 8.0. This can be attributed mainly to the buffering system of limestone. It was concluded that the precipitation of metals is governed by the solubility product. Since all effluent guidelines require an effluent pH between 6 and 9, the use of carbonate treatment is, therefore, recommended because of its buffering capacity value which is around pH 7. Still CaO provides substantial precipitation efficiency and an economic mean of treatment. The disadvantage is the difficulty to control the pH of the liquid waste. However, this problem could be overcome by using acid to control the pH if needed.

Keywords: Heavy metals, Removal, Chemical coagulation, Industrial wastewater, Precipitation and co-precipitation, Copper, Nickel and Manganese.

Heavy metals are among the most hazard pollutants that affect both the environment and man health as well. Several sources of heavy metals in the environment are anthropogenic in nature ⁽¹⁾. Besides, heavy metals content in

non-polluted soil is largely depending on the rocks from which the soil parent materials were driven. Meanwhile, the weathering process is quite relevant⁽²⁾. In this respect, heavy metals can have a detrimental effect on organisms in the environment as well as on humans who ingest them.

The presence of metals in trace amounts in the environment is fundamental. Some metals such as Fe, Mn and Zn are considered essential elements for plant growth and thus play an important role in the biochemical cycle⁽³⁾. On the other hand, some metals such as Hg, As, Cd and Pb are considered very toxic to plant⁽³⁾. For instance, early anti-fouling paints used mercury as a base. It was not until the public health tragedy of mercury contamination of Minimata Bay, Japan, in the 1950's that anybody even became aware of the problems caused by heavy metals in the aquatic environment⁽⁴⁾. It was at this point that there was a switch from mercury to tributyl-tin (TBT) as the base for anti-fouling paint. However, switching to TBT did not resolve all of the problems having to do with heavy metals in bottom paint⁽⁵⁾.

Man has set up complex treatment processes to prevent or control pollution from wastewater reaching the environment. The principle objective in wastewater treatment is to eliminate or reduce contaminants to levels that cause no adverse effects on humans or the receiving environment^(6,7). A common method of removing heavy metals from wastewater has been to mix it with sewage, where conventional primary, secondary and tertiary treatment would then remove heavy metals. However, secondary and tertiary processes require high input of technology, energy and chemicals^(8,9). The costs, maintenance and operation of such technology are of great concern^(7,10). Such treatment processes should, therefore, be very attractive and economically justifiable for large-scale treatment plants, especially in cash-strapped third world countries. A cheaper, but efficient treatment technology was therefore sought such as oxidation ponds, constructed wetlands and trickling filter^(11,12).

It is worth mentioning that heavy metals are ubiquitous in natural waters at low concentrations ranged between ppb and ppm⁽¹³⁾. Due to their refractory nature, their bioaccumulation in the food chain and potential toxicity, heavy metals may have an adverse impact on the environment⁽⁴⁾. As result a great concern has been arising in the last few decades stressing the need for effective wastewater treatment methods for metals removal^(1,14,15).

Metals exist in wastewater in many forms, including soluble, insoluble, inorganic, metal organic, reduced, oxidized, free metal, precipitated, adsorbed and in complex formation⁽¹⁾. Treatment processes for metals removal must be selected according to the existing form of the metal. Otherwise, the metal must be converted to a suitable form compatible with the removal process^(16,17). In general, to be removed from wastewater, metals must be precipitated or otherwise attached to an insoluble form through adsorption or ion-exchange⁽¹⁷⁾. A number of soluble metal ions are simply precipitated on addition to

wastewater⁽⁶⁾. But this natural removal mechanism is most effective at high metal concentrations and is usually not sufficient to reduce metals to the levels required by water quality standards or safe reuse^(7, 18).

The objective of the present investigation is to study the effect of different precipitants and chemical coagulants on the removal of Ni, Cu and Mn from the industrial wastewater. Therefore, it is found essential to select the food industrial wastewater because it contains high level of suspended solids of plant origin as organic bio-waste. Level of heavy metals in this wastewater was also determined. Evaluation and correlation between different chemical coagulants for metals removal are also the aim of this work.

Material and Methods

The industrial wastewater of a selected food company was used in this study. Reagent grade soluble salts of $\text{Ni}(\text{SO}_4) \cdot 6 \text{H}_2\text{O}$, $\text{Cu}(\text{SO}_4) \cdot 5\text{H}_2\text{O}$ and $\text{Mn}(\text{SO}_4) \cdot \text{H}_2\text{O}$ were added to this wastewater to provide 10 mg/l of each metal as an artificial contaminated industrial liquid waste. Reagent grade concentrated H_2SO_4 was used to decrease the pH of the wastewater to range of 2.0 through the entire study. The investigated precipitants, coagulants and coagulant aids are: sodium hydroxide, FeCl_3 , alum, lime (CaO) and limestone (CaCO_3). All the chemical coagulant solutions were freshly prepared prior experimentation.

The "Jar Test" procedure, as devised and modified by Culp and Culp⁽¹⁹⁾ was used in all experiments. One of these "Jars" was kept as the "control one" without adding any chemical treatment. The precipitants were added during the flash mixing process at the speed of 400 rpm for a period of 1 min followed by flocculation at the speed of 40 rpm for a period of 20 min. The mixtures were left 30 min for settling⁽¹⁹⁾. The residual metals were determined in the supernatant of each jar.

The concentration of nickel, copper and manganese was determined in the acidified samples. The acidification was carried out by concentrated nitric acid to pH below 2.0⁽¹⁶⁾. For determination of heavy metals atomic absorption spectrophotometer "Instrumental Laboratories" model (551) equipped with heated Graphite Atomizer Model (651) and deuterium background corrector was used for this purpose.

Meanwhile, the physical and chemical characteristics of the food industrial wastewater was determined monthly over a period of twelve months. All analytical procedures were carried out according to the Standard Methods (APHA)⁽²⁰⁾.

Results and Discussion

The physical and chemical characteristics of the industrial wastewater are given in Table 1. The pH of this wastewater ranged from slight alkaline to neutral and it was highly turbid at an average of 1040 NTU. The total solids were high and the total suspended solids were moderate at the average of 1632 and 485 mg/l, respectively.

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The electric conductivity ranged from 600 to 1000 $\mu\text{mhos}/\text{cm}^{-1}$. The COD and BOD were within the high range of 4875 and 4168 mg/l, respectively. The nitrates, organic nitrogen and chlorides were within the low range at the average of 15.8, 14.4 and 98 mg/l, successively. However, the phosphates and sulfates were slightly high at the range of 92.6 and 77 mg/l, respectively. The average background concentration of copper, manganese and nickel was 0.09, 0.71 and 0.11 mg/l, successively (Table 1).

TABLE 1. Physical and chemical characteristics and metals concentration of the selected food industrial wastewater (from period of 12 months).

Parameters	Min.	Max.	Mean Value
pH	5.7	7.5	6.5
Temp.	14.4	23.9	19.2
Turbidity (NTU)	900	1100	1040
E.C. ($\mu\text{mhos}/\text{cm}^{-1}$)	600	1000	780
Total suspended solids (mg/l)	246	664	485
COD (mgO_2/l)	1267	6000	4875
BOD (mgO_2/l)	1380	8360	4168
Nitrates mg/l	9.1	29.0	15.8
Total Phosphates ($\text{mg PO}_4^{3-}/\text{l}$)	50.5	173.5	92.6
Organic Nitrogen ($\text{mg N}_2/\text{l}$)	10.2	20.1	14.4
Sulfates (mg/l)	48	124	77
Chlorides (mg/l)	42	121	98
Total solids (mg/l)	908	2093	1632
Metals			
Copper (mg/l)	0.06	0.14	0.09
Manganese (mg/l)	0.65	0.89	0.71
Nickel (mg/l)	0.10	0.16	0.11

Sodium hydroxide (NaOH) at different pH

By using sodium hydroxide as precipitant, the results obtained (Fig. 1) showed that increasing the pH value increased metals precipitation. The optimum pH for Cu, Ni and Mn was 9.5 at which removal reached 97.5% 96.0%, and 90.0%, respectively. Increasing the pH value to 12 increased the removal of Mn to 93%. Figure 1 represents the decrease of metal concentration at pH 9.5 from 10.0 mg/l down to 0.25, 0.40 and 1.0 mg/l for Cu, Ni and Mn, respectively. At pH 12.0, removal of all the studied metals reached 100% residual concentration of Cu, Ni and Mn was further decreased down to zero mg/l.

Ferric chloride (FeCl₃) in combination with sodium hydroxide at different pH

The use of ferric chloride (FeCl_3) at different pH values was also investigated. Sodium hydroxide was used to cover the pH range between 2.0 and 12.0 along with the addition of 50 mg/l ferric chloride; that is equivalent to 17.22 mg Fe^{+3}/l ; at each point. The results (Fig. 2) indicate that the removal efficiency was improved noticeably. At pH 7.5 the removal of Cu and Ni reached 95% each, while Mn was removed only at 81%. The residual concentration of Cu and Ni

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was 0.5 mg/l each and was 1.9 mg/l for Mn. The optimum pH was 9.0. The removal rate of all the studied metals increased up to 95% at which the residual concentration of each metal was 0.5 mg/l. Further increase of pH to 12 increases the removal of all metals up to 98% at which the residual concentration of each metal was further decreased down to 0.2 mg/l (Fig. 2). Iron will be precipitated with the other metals, because FeCl_3 here is a co-precipitant. Sodium hydroxide is the main agent that forms the hydroxides of metals here.

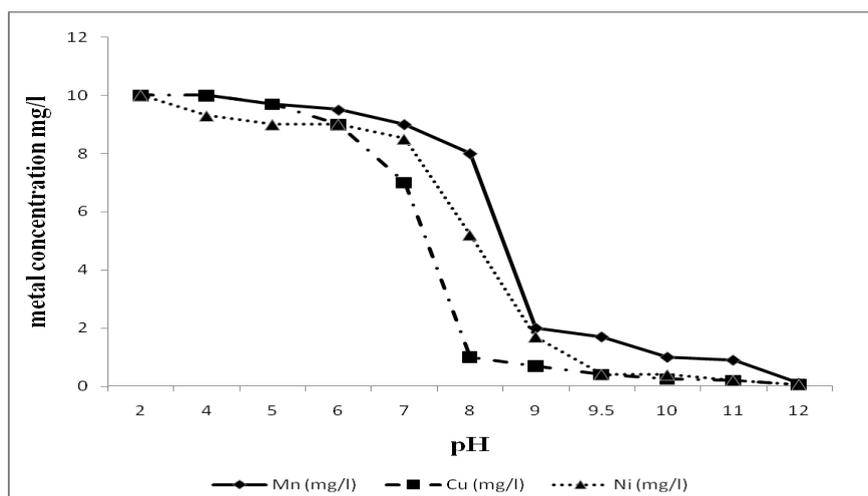


Fig. 1. Effect of NaOH at different pH values on the precipitation of Mn, Cu and Ni in artificial contaminated industrial wastewater.

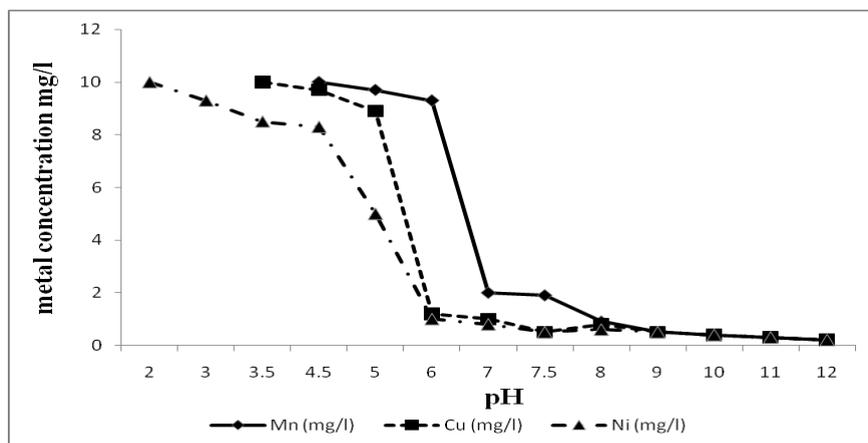


Fig. 2. Effect of NaOH combined with FeCl_3 at different pH values on the precipitation of Mn, Cu and Ni in artificial contaminated industrial wastewater.

Alum in combination with sodium hydroxide at different pH

The use of alum as $[\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{H}_2\text{O}]$ at different pH values ranging from 2.0 to 12.0 was also carried out. Sodium hydroxide was further used to adjust the pH values at a constant alum dosage of 70 mg/l, which is equivalent to 2.17 mg Al^{+3} /l. The results (Fig. 3) indicate that optimum pH value was 9.5 at which 98% of Cu, 96% of Ni and 84% of Mn were removed. Residual concentration of metals was decreased down to 0.2, 0.4 and 1.6 mg/l for Cu, Ni and Mn, respectively. Increasing the pH up to 12.0 the removal rate of Cu and Ni reached 100% (*i.e.* residual concentration zero mg/l). However, the removal rate of Mn did not exhibit any further increase and was still at 84%. Aluminum will be precipitated with the other metals, because alum here is a co-precipitant. Sodium hydroxide is the main agent that forms the hydroxides of metals here.

The use of lime at different doses

Further study was carried out to investigate the use of lime (CaO). Different doses of lime were added to wastewater ranging from 50 to 300 mg/l. The study covered the range of pH from 2.0 up to 12.0 according to the addition of lime dose to the wastewater. Remarkable improvement in the precipitation system was demonstrated by increasing the pH value (Fig. 4). At 220 mg/l lime the pH reached 9.0 at which over 98% of Cu and Ni each was removed. The residual concentration decreased to 0.2 mg/l each. At this point, Mn removal reached 93% and the residual concentration was 0.7 mg/l. By increasing the pH to 10.0, the optimum removal of Mn was achieved namely 96% with a residual concentration at 0.4 mg/l. Increasing the pH to 11.0 increased the removal of Cu and Ni up to 100%, while the removal rate of Mn did not show any further improvement (Fig. 4).

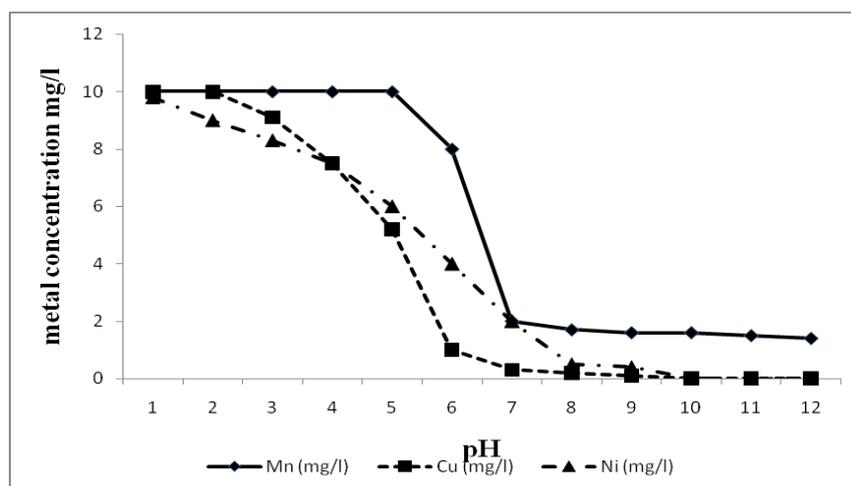


Fig. 3. Effect of NaOH combined with Alum at different pH values on the precipitation of Mn, Cu and Ni in artificial contaminated wastewater.

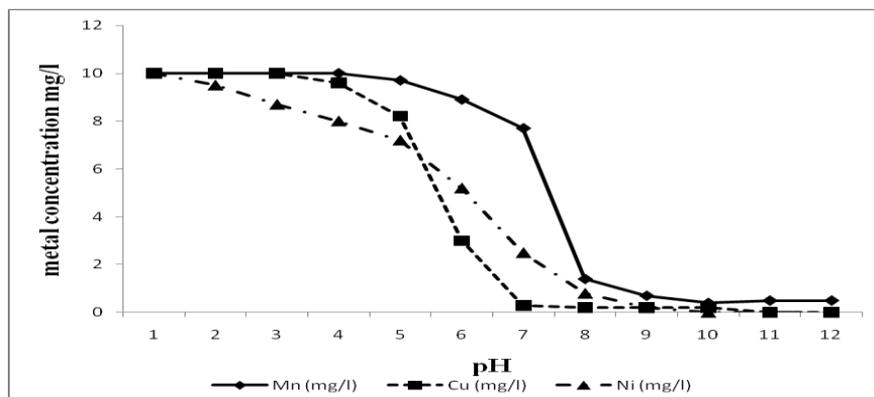


Fig. 4. Effect of different lime (CaO) doses on the removal Mn, Cu and Ni in artificial contaminated wastewater.

The use of limestone at different doses

Use of limestone (CaCO_3) at different doses covering the range from 0.2 g/l to 5.0 g/l was investigated. Results obtained (Fig. 5) showed that increasing the CaCO_3 dose from 100 to 2000 mg/l led to a slow increase in the pH value up to 6.5 (Table 2). Further addition of CaCO_3 up to 4.0 g/l exhibited very slow increase of the pH up to 7.5 which is attributed to carbonate buffering capacity system. The removal rate of the metals studied (Fig. 5) showed that use of 1.0 g/l raised the pH to 5.85 and the removal of Cu, Ni and Mn to 100, 90.2 and 75.1%, respectively. By increasing the CaCO_3 to 3.0 g/l, the pH reached 7.0 at which 100% of Cu and Ni each was achieved. At this point Mn removal reached 90.6% (*i.e.* residual concentration was 0.94 mg/l, Fig. 5). One significant benefit of the CaCO_3 is the buffering effect provided by both carbonate and bicarbonate ions. Table 2 records the gradual change in the pH of the wastewater according to the added dose of limestone.

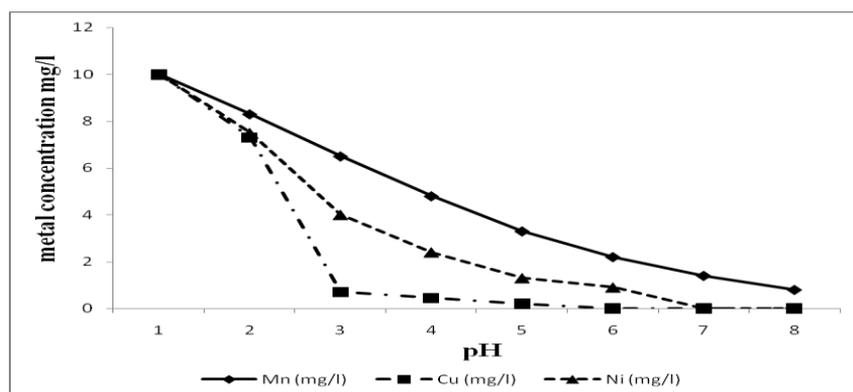


Fig. 5. Effect of different doses of lime (CaO) on the precipitation of Mn, Cu and Ni in artificial contaminated water.

TABLE 2. Effect of different calcium carbonate (CaCO₃) doses on the pH of the artificial contaminated industrial wastewater.

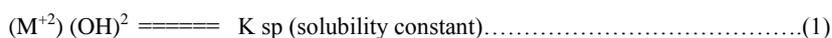
Limestone (CaCO ₃) dose (mg/l)	pH value of the wastewater
0.0	2.0
100	3.0
250	4.3
500	5.0
1000	5.85
2000	6.5
3000	7.0
4000	7.5
5000	8.0

Correlation between the efficiency of the studied chemical coagulants on the removal of Cu, Ni and Mn at the optimum pH is given in Table 3. This correlation shows that the examined chemical coagulants were all efficient in the removal of the studied metals. Between 95 to 100% of Cu and Ni could be removed. However, Mn was less removed within the range between 90 to 99%. The highest efficiency of chemical coagulants was exhibited by lime and limestone. Nevertheless, the later required high dose between 3000 or 5000 mg/l to reach pH 7.0 and 8.0, respectively. On the other hand, lime exhibited high efficiency of metals removal at pH 10.0 (Table 3). Still, lime removal efficiency at pH 9.0 was 98.0% for Cu and Ni each and 93.0% for Mn.

TABLE 3. Correlation between the efficiency of the studied chemical coagulants on the removal of Cu, Ni and Mn at the optimum pH.

Chemical coagulant	Optimum pH	% of metal removal		
		Cu	Ni	Mn
NaOH	9.5	97.5	96.0	90.0
FeCl ₃ + NaOH	9.0	95.0	95.0	95.0
Alum + NaOH	9.5	98.0	96.0	84.0
Lime (CaO)	9.0	98.0	98.0	93.0
	10.0	98.0	99.0	99.0
Limestone (CaCO ₃)	8.0	100	100	96.0
	7.0	100	100	90.6

It can be concluded that the co-precipitation of metal hydroxides is governed by the concentration of metal ion in solution and the pH value. As the pH increases, the co-precipitation of metal hydroxide increases. However, certain amphoteric metals will re-dissolved at high pH^(16, 21). This relationship is mostly governed by the following solubility product equation:⁽²¹⁾



The solubility product constants for a number of metals have been already published⁽²¹⁾. However, because of precipitates aging, incomplete solid separation, or co-precipitation and adsorption effects in wastewater, solubility products provide only general guide to residual metals concentration to be expected in practice^(22, 23).

The overall results indicate that the optimum pH for hydroxide precipitation of the studied metals is varied between pH 6.5 and 11.0. Since all effluent guidelines require an effluent pH between 6 and 9, the use of carbonate treatment is, therefore, recommended because its buffering capacity value is around pH 7. However, lime (CaO) provides substantial precipitation capacity and an economic mean of treatment for heavy metal removal. The disadvantage is the difficulty to control the pH of the liquid waste. To get over such disadvantage acids and NaOH could be used to control the required pH if needed. The sludge formed could be handled as hazardous chemicals since it contains heavy metals. However, the most important is that heavy metals were removed from the wastewater that could be reused safely without any hazard to man.

Conclusion

Removal of metals from liquid waste can be achieved efficiently by increasing the pH value of treatment system. Chemical coagulation including lime (CaO), limestone (CaCO₃) and perhaps sodium hydroxide is effective in the precipitation and / or decreasing the solubility of heavy metals in the industrial wastewater.

A major environmental variable that determines the mobility of most metals in water is pH. As pH drops below approximately pH 10 or 11, most metals increase their solubility in water exponentially with the exception of carbonate precipitation. At high pH and low solubility, metals may either precipitate out of solution or bind to solid particles in the adsorption process.

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ازالة المعادن الثقيلة من المخلفات الصناعية السائلة باستخدام المجاطات الكيميائية

حسين إبراهيم عبد الشافى

قسم بحوث تلوث المياه – المركز القومى للبحوث – الدقى – القاهرة – مصر.

تتناول هذه الدراسة ترسيب المعادن من المخلفات السائلة باستخدام المجاطات الكيميائية وتمت هذه الدراسة على المخلفات السائلة الخاصة باحدى شركات الصناعات الغذائية – حيث أضيف إليها مخلوط من أملاح كبريتات النحاس، النيكل والمنجنيز بتركيز 10 مجم / لتر لكل معدن – تم اختبار تأثير إضافة جرعات مختلفة من المجاطات الكيميائية وتأثيرها على ترسيب، وإزالة هذه المعادن من المخلفات السائلة. فتم اختبار المجاطات الكيميائية الآتية: صودا كاوية ، مخلوط الصودا كاوية مع 50 مج /لتر من كلوريد الحديدك ، مخلوط الصودا كاوية مع 70 مج /لتر من الشبة ، الجير ، الحجر الجيري .

دلت النتائج عن فاعلية المعالجة الكيميائية لترسيب المعادن المختبرة بنسب عالية تتراوح بين 90 % الى 100. فإضافة 220 مجم/ لتر من الجير وصلت نسبة ازالة النحاس الى 98% وعند زيادة الأس الهيدروجيني الى 8,5 وصلت نسبة ازالة كل من النحاس، النيكل والمنجنيز الى أكثر من 94% ، وعند إستخدام 3 جرام/ لتر من الحجر الجيري وصلت نسبة ازالة جميع المعادن المختبرة الى حوالى 96% ، وبإضافة مخلوط الصودا الكاوية مع 70 مجم / لتر من الشبه زادت نسبة الإزالة الى 98% لكل المعادن المختبرة حيث يستنتج من هذه الدراسة أن المجاطات الكيميائية لها قدرة فعالة فى ترسيب وإزالة المعادن من المخلفات السائلة ويمكن إستخدامها فى الصناعة لهذا الغرض لحماية المسطحات المائية من التلوث بالمعادن الناتجة من قذف هذه المخلفات السائلة.