

# Review of Purification of Industrial Wastewater

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**Abstract** - There has been an excessive increase in industrial wastewater due to all the industrial and commercial activities. Purification of industrial wastewater involves all the various techniques and processes used to treat wastewater and bring down the concentration of the pollutants to a permissible limit. In this paper, the various methods used for removal of those contaminants are reviewed. The contaminants that are dealt with in this paper are Pyridine, Chromium and Chromate ions, Phenols along with Phenoxides and Chlorophenols, Titanium and Arsenic. Pyridine removal can be done by adsorption at high area C-cloth electrodes using in-situ optical spectrometry and advanced oxidation processes. Phenols and its derivatives can be removed by using the process-adsorption and electrosorption which are felt at C-felt electrodes and also by adsorption of phenol using cereal by-products. Chromium removal can be done by Solvent Extraction or by using solid olive waste to adsorb chromate ions. Arsenic is removed by adsorption and precipitation. The various separation procedures aim towards complete or partial removal of the pollutants and are discussed in detail in the paper. Conclusions are drawn and the most economical and efficient method was listed over.

**Keywords**— Arsenic; Chromium; Phenols; Purification; Pyridine; Wastewater

## I. INTRODUCTION

Wastewater is the water which is highly polluted by any anthropogenic sources which has led to deterioration in its quality and made it unfit for any kind of human use. Most of these anthropogenic sources consists of industrial effluents, domestic and sewer wastes, agricultural wastes and surface run offs. These are present in levels which are beyond permissible limits and they render the water to be polluted. As a result, concentration of various chemicals increases considerably in the water due to its constant exposure to different anthropogenic sources. The heavy metals present in the water like Arsenic, Cadmium, Lead not only degrade the quality of any water body if present in excess quantities but also cause serious harm to aquatic and human life. Exposure to such toxic metals is carcinogenic and can result in deadliest of diseases. According to Babel and Kurniawan, 2003, Arsenic is known to cause visceral cancer and several vascular and skin diseases and Chromium is mentioned to cause nausea, vomiting, diarrhoea and headache and is also carcinogenic.[1] Pyridine and Phenols are also known to cause several health ailments.

Removal of these contaminants from the waste water is of paramount importance not only to purify the water but also to save much aquatic and human life which is directly or

indirectly related to it via the food chain. There may be various methods to remove the contaminants which are devised from time to time depending upon the needs of the civilisation. Pyridine and Phenolic derivatives both can be removed by Adsorption at high area C-cloth electrode. Oxidation and natural adsorbents can also be used to remove them respectively. Solvent Extraction and Adsorption using natural adsorbents are highlighted methods for chromium and chromate ions. Whereas, titanium can be removed by catalytic electrolysis and membrane separation methods. Arsenic, a heavy metal can be removed by adsorption followed by precipitation. The various methods are listed in detail and a reviewed study is done on the same.

## II. CONTAMINANTS TO BE REMOVED

The following contaminants are to be removed from the industrial wastewater in the following mentioned way:

### A. Pyridine

Pyridine (Py) is a very important component which is used in chemical as well as medical industries and also in agriculture. Since it is highly soluble in water, it is found mostly in aquatic environments. There are a lot of industries such as dyes, herbicides, shale oil processing etc. from where in pyridine is released. Being one of the major pollutants in waste water from several industries, removal of pyridine is of prime importance as it has harmful effects on health. The following processes are used to remove pyridine:

1) *Adsorption at high area C-cloth electrodes using in situ optical spectrometry*: In this process high area carbon cloth (C-cloth) electrodes are used as quasi 3-D interfaces, coupled with in situ UV-Vis spectrophotometric techniques. Py is then completely removed by anodic polarization. Experiments were carried out by J. Niu et al. (2002) at 25°C in 0.01M electrolyte solutions. It not only provided adequate conductivity for polarization experiments but also for suppression of possible effects of pH changes on adsorption. A non-buffered solution Na<sub>2</sub>SO<sub>4</sub> and another HOAc+NaOAc buffer were used for this experiment.[2]

The present examination done by Niu and Conway shows that Py is the one of the commonly occurring organic pollutants in industrial waste water which can be completely removed by adsorption on high area C-cloth electrodes. Moreover, electrical polarization mostly enhances the removal efficiency. The polarization electrosorption process was performed both potentiostatically and galvanostatically. The

latter is preferred as it gives large removal rates and is also quite convenient. Also anodic galvanostatic polarisation gives stronger electrosorption effects as compared to cathodic polarization. With the increase of the C-cloth electrode area, the rate of adsorptive removal of Py increases. The electrolyte has a very important role in the removal of pyridine. The pH changes are eliminated by using HOAc+NaOAc buffer solution.

2) *Advanced oxidation processes (AOP)*: Fusing is done to remove silica from the sample. 1 gm of the slag sample is accurately weighed in a platinum crucible. As this process is aimed at the conversion of contaminants into inorganic and harmless products, it is widely used for keeping a check on the pollution.[3]

In the treatment by AOP by BC Bag et al. (2009), the generation of hydroxyl radicals which are of utmost importance, is the first step, and they react with organic pollutants which results in a degraded product. There are different processes of generating the hydroxyl radicals, namely,  $H_2O_2$  photolysis ( $H_2O_2/UV$ ), Fenton's process ( $H_2O_2/Fe^{2+}$ ), photo-assisted Fenton's process ( $H_2O_2/Fe^{2+}(Fe^{3+})/UV$ ), photo-catalysis ( $TiO_2/h\nu/O_2$ ) etc. In all spheres, the AOP was used either for a pure compound or in case of a one component system.

The degradation of pyridine was carried out by four AOP's namely, UV/  $H_2O_2$ , Fenton's and photo-Fenton's processes and photo-catalytic ( $TiO_2/h\nu$ ) processes. In the photo-Fenton's process a maximum of 74.0% COD removal was obtained at a pH 2.0. The rate of COD removal was in the order of  $H_2O_2/Fe^{2+} > TiO_2/ H_2O_2 > H_2O_2$  (without catalyst)  $> TiO_2$ . pH affects the COD removal. At lower pH (<4.0), the rate of degradation and removal of COD was higher, whereas only 6% increase in the COD removal was obtained with the decrease of pH from 4.0 to 2.0. When the degradation of the waste solution was carried out with  $TiO_2$  and without  $H_2O_2$  no significant effect of pH on COD removal was obtained.

### B. Phenols, phenoxide and chlorophenols

Phenolic compounds are considered to be one of the major pollutants in industrial waste water. Being metabolites of some pesticides, they cause significant pollution of waste waters. They are present in wastes from the manufacture of formaldehyde resins, lacquers and binders, pharmaceuticals and pesticides, from coking and coal distillation plants, and in soil and vegetable residues. Chlorophenols and nitrophenols, which are used in industry and agriculture, and as wood preservatives, can also be present in raw water as a result of spillage or accidents.

1) *Adsorption and electrosorption at high-area C-felt electrodes*: The destruction of these components can be brought about by anodic oxidation while phenols can also be treated by  $TiO_2$ -based photooxidation-catalysis. The adsorption and electrosorption of pollutants by in situ UV spectroscopy at high area C-felt electrodes have recently emerged. It was seen that the initial concentrations (about 1 mM) of these compounds can be reduced by a factor of 3 or more from waste-waters by adsorption at the C-felt. Also, the rate of adsorption of these compounds can be enhanced by

polarizing the C-felt electrode, in the presence of an inert supporting electrolyte such as  $Na_2SO_4$ . [4]

According to the experiments carried out by Ayranci and Conway (2001), PhOH was observed to have absorption spectra bands at 210 and 269 nm. Conversion of PhOH to  $PhO^-$  anion takes place. PhOH and  $PhO^-$  anions can hydrolyse in aqueous solutions. Determination of concentration of PhOH, in situ, spectrophotometrically during adsorption at the C-felt was done based on its absorbance at 269 nm. Absorbances at 269 nm were converted into concentrations. It is seen that the 0.82 mM initial concentration is reduced to about 0.25 mM, i.e. by a factor of more than 3, within 2 hours. Pre-wetting of the C-felt in pure water prior to adsorption had been found to enhance the adsorption rate considerably by almost a factor of two.

There are two consecutive periods of positive and negative polarization. In positive polarization, a change of the PhOH spectrum to that of  $PhO^-$  took place. The adsorption of  $PhO^-$  is faster than that of PhOH due to positive charging of the C-felt surface. During negative polarization, the spectrum returned to that of PhOH due to Faradic regeneration of  $H^+$  ions or consumption of  $OH^-$  ions.  $PhO^-$  on one hand begins to be desorbed due to negative polarization of the surface, while on the other hand PhOH concentration remains almost steady. Considerable amounts of PhOH and  $PhO^-$  are adsorbed on open-circuit at the C-felt. The solution concentration of phenolic species decreases to a very low level just by allowing adsorption to continue on open-circuit.

Also there is a faint enhancement in the rate of adsorption upon positive polarization. PhOH is the main adsorbing species here since  $PhO^-$  ion concentration is quite weakened by hydrolysis towards the PhOH side rather than by adsorption of the  $PhO^-$  ion. Because of hydration of the phenoxide anion, the electrosorption of  $PhO^-$  at the C-felt is much less than that of PhOH. The phenoxide anion is more hydrophilic than PhOH, with the  $-O^-$  function causing water to be bound in the anion's hydration shell.

The presence of  $Na_2SO_4$  was essential for allowing the passage of a higher current of about +2 mA at the C-felt than that possible in its absence which is about +0.6 mA. There was no effect on adsorption and the concentration decreased by more than half of its initial value within an hour of the open circuit adsorption.

The adsorption behavior of the three monochlorophenols is different. The increasing order of rate of adsorption is 4-<3-<2-chlorophenol. This shows greater extents of adsorption for 3- and 2- chlorophenol than for 4-chlorophenol, but do not differentiate clearly the behavior of the 3- and 2-chlorophenols.

Phenol, phenoxide and chlorophenols that constitute an important class of organic pollutants in industrial waste-waters can be removed to substantial extents by adsorption and electrosorption at high-area C-felt electrodes. The adsorption/electrosorption processes can be followed quantitatively by in situ UV spectroscopy. Pre-wetting of C-felt and the use of a supporting electrolyte such as  $Na_2SO_4$  in the adsorbate solution enhances the adsorption. The rate of adsorption of  $PhO^-$  is increased when the C-felt surface is positively polarized.

2) *Adsorption of phenol using cereal by-products as a new adsorbent*: The elimination of phenol from wastewater can be done by synthesizing aqueous phenol solutions and carrying out adsorption on a non-activated carbon which has been prepared from cereal by product on laboratory scale and hence studying its characteristics.[5]

In general, phenolic wastewater treatment is divided into two basic categories:

A destructive process (where destructive oxidation takes place using oxidising agents like ozone, hydrogen peroxide, magnesium oxides etc.) and a recuperative process like adsorption, membrane separation and solvent extraction.

Experiments were carried out by Sihem and Lehocine (2012), where a known quantity of solid material (prepared from cereal by product) was mixed with a phenol solution of known concentration. The mixture was then agitated of a known time duration at a fixed temperature and subsequently filtered on filters micro pores. The filtrate was collected and its pH was measured along with the analysis of the filtrate by UV-Visible Spectrophotometer and the adsorption capacity were then analysed.

Subsequently, different parameters such as time of contact, pH and initial concentration were noted and their effects were studied.

In context to the effect of contacting time, it was observed that equilibrium is attained at the end of 90 minutes. The process is rapid at the start but gradually slows down because of the unavailability of the free active sites as they are reduced with the advancement of time.

As far as the study of kinetic models is considered, different models were tested and based on the results it was concluded that the process is most precisely represented by second order kinetics indicating chemisorption. Diffusion intra particle model follows next.

pH also determines the rate of retention. At low pH of 2 the retention output is maximum and it decreases gradually becoming minimum at pH of 8 the reason behind this is that low pH has a consequence of lowering of the negative charge on the adsorption surface thus facilitating the adsorption of phenol ion which is over all positively charged.

During the study of adsorption isotherms, it was observed that a multi-layer isotherm with steps is obtained which does not go in accordance with any of the traditionally available isotherms. Still in comparison with all other existing isotherm this phenomenon is represented best by an isotherm with multi-layer (BET) which gives a factor of correlation of 0.99975.

In a nut shell, this study shows that cereal by product an abundant natural material can be used effectively and efficiently for the removal of phenol from waste water.

### C. Chromium

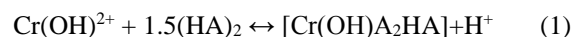
The environmental impact of the emissions of heavy metals present in the industrial effluents is ever increasing. It imposes risks of harmfulness with respect to the living organisms. Chromium comes from the baths of tannery, galvanoplasty, metallurgical industry, textile.

### 1) Solvent Extraction or Liquid Membrane method:

The solvent extraction is largely used. It is profitable for recovery, the separation and the purification of metals. The extraction of trivalent Chromium is done in liquid-liquid phase because it is present as traces in many industrial effluents. The mechanisms associated with this are exchangers of cation like the phosphine acid di(2,4,4-trimethylpentyl) (Cyanex) and exchangers of anions such as Primene JMT, the Tri-n-octylamine.[6]

According to the experiments conducted by Hassaine-Sadi and Sadoun (2005), extraction of trivalent chromium by the acid di(2,4,4-trimethylpentyl)phosphonic (cyanex) for the extraction of Chromium(III) in sulphuric medium is done by examining the influence of the parameters of extraction. Outputs of extraction reach a maximum of about 80% in Chromium(III) for chloroform. An improvement of the extraction percentage of Chromium(III) by the cyanex is obtained which passes from 75% (in the kerosene) to 80% to 0.1 M H<sub>2</sub>SO<sub>4</sub> in chloroform. pH also effects extraction significantly. By increasing the pH, until at pH equal to 6, a stage is obtained where the extraction of Chromium (III) becomes constant, the order of 80%.

Chromium(III) in aqueous solution is extracted in a hydrolyzed form. The extraction of the chrome(III) by the acid di(2,4,4-trimethylpentyl) phosphonic is done by a mechanism exchanges cations according to (1):



Extraction of trivalent chromium was also done by the primene (JMT). pH has a very significant effect on the extraction, for values of pH ranging between 0 and 2, the extraction increases and reaches a maximum from approximately 45% with pH of 2 and the percentage decrease is about 18% for a pH of 6. For the diluted solutions Chromium(III), the extraction by primene JMT is significant, but for the strong initial concentrations in Cr(III), the percentage of extraction are weak, therefore it ends up at the saturation of organic phase. The temperature was also varied and a substantial improvement of the extraction of Chromium(III) is noted The extraction passes from 13 to 58% when temperature is increased from 25°C to 50°C for a concentration of 0.1 M in sulphuric acid.

The extraction by the acid di(2,4,4-trimethylpentyl) phosphonic showed that the extraction percentage are about 80% with equal pH 6 in the kerosene. The extraction is done according to a mechanism of exchange of cations, the extracted species has the form: [Cr(OH) A<sub>2</sub>HA]. By using Primene-JMT (RNH<sub>2</sub>) and the Tri-n-octylamine (R<sub>3</sub>N) for the extraction of Chromium(III), the extraction follows a mechanism of extraction of exchange d'anions, the extracted species are respectively in forms: [RNH<sup>3+</sup>, (CrSO<sub>4</sub>)<sup>2-</sup>] and [R<sub>3</sub>NH<sup>+</sup>, (CrSO<sub>4</sub>)<sup>2-</sup>].

### 2) Solid olive waste used to adsorb chromate ions

This gives a detail on how solid olive waste can be used to remove chromate ions from the solution and the solid olive which is a waste would also get utilised by converting it into activated carbon. Studies were carried out on metal ion adsorption and desorption by Amer El-Hamouz et al. (2007) which are discussed below.[7]



Initially experiments were conducted where activated carbon was added to aqueous solutions of  $K_2Cr_2O_4$  and the remaining aqueous ion concentration was measured using auto calibrated atomic adsorption spectrometer. Samples were taken and under controlled pH, different parameters such as particle size and ion concentration were studied.

Further, experiments on desorption were also conducted where the ability of pre-treated AC to desorb chromate ion was studied. Here, already chromium treated AC was placed in a chromate free solution for around 2 hours to allow desorption.

Overall, from these experiments chromate ion adsorption using AC is studied. Equilibrium saturation was obtained after 2 hours of constant shaking and the maximum adsorption uptake was observed to be 1.2 mg/gm which is comparable with the available literature.

Also, particle size effect was studied where particle in the range of 400-850  $\mu m$  was found to be most effective. Also, in accordance to the available literature, it was observed that the relative chromate uptake value is higher for smaller particles as they have relatively higher external surface area. Also it was observed that in large particles, there was not much effect of particle size on the adsorption of chromate ions. This can be explained by understanding the concept of micro pores where it was seen that most of the adsorption occurs in the micro pores which does not necessarily depend upon the particle size and hence external surfaces do not actually contribute in any manner.

Subsequently, it was observed that the chromate uptake is higher for higher pH as chromate ion adsorption on AC increases when acidic ions are removed from the surface hence contributing to higher pH. The most effective pH range was observed to be 5-6. It can be questioned that low pH would have enhanced the uptake of negative charged species as they are of opposite charges, but the appropriate reason here is that at lower pH chromate ions exist in the form of a mixture of  $HCrO_4^-$  and  $Cr_2O_7^{2-}$  ions and these species in the presence of activated carbon result to a positive charged Cr(III) ions which would result in a repulsion b/w the positive groups.

To enhance the potential value of AC, chromate desorption from used AC should also be studied. It was seen that most of the desorption occurs at lower pH values as lower pH results in the formation of Cr(III) ions which do not adsorb into the AC micro pores resulting to desorption.

So finally, the experiments and the subsequent studies show that following a processing cycle that combines adsorption at AC at higher pH value and subsequent desorption from ac at lower pH values would help to remove chromate ions from the solution to a large extent. The main advantage of this process is that it removes organic material from solid surfaces which results to relative high surface area which acts as a boon for removal of chromate ions.

#### D. Tritium

A wastewater stream from a nuclear plant contains tritium along with some solid waste particles. Tritium is a radioactive isotope of water,  $^3H$ , which causes little hazard or risk at low concentrations and in gaseous form. At higher concentrations, tritium poses a more serious threat,

especially when suspended in water, because it can be ingested or absorbed into the body easily.

#### 1) *The combined electrolysis catalytic exchange*

*method:* This process utilizes wetproof catalyst and is extremely useful for extracting tritium from water due to its high separation factors and near-ambient operating conditions. As stated by I.A. Alekseev et al. (2001), this process is regarded as an alternative for detritiation in comparison to conventional water distillation (DW) and vapour phase catalytic exchange (VPCE) processes in the ITER isotope separation system. [8]

A chemical exchange column is used which is filled with alternating layers of wetproof catalyst and stainless steel spiral-prismatic packing. The column consists of three separation sections connected through a distributor of liquid. Each section is provided with a heating water jacket for maintaining the column temperature. The wetproof catalyst used consists of 0.8 wt.% platinum deposited on porous polysorb (styrene-divinylbenzene copolymer). Tritium injected to the tower had a feed flow of 108 Bq/kg and the tritium concentration in the top product is less than 105 Bq/kg. The samples of heavy water were analysed by the IR-spectrophotometer, and gas samples by gas chromatography. Tritium was measured by the liquid scintillation method. The tests of the plant have shown a high efficiency of isotope separation.

2) *Membrane Separation Method:* In this process, a 13-stage recycling cascade polyphosphazene membrane process was designed to separate tritium from water. The membrane separation process is designed to remove 95.00% of the tritium into permeate, with 99.90% of the flow as EPA (Environmental Protection Agency) approved clean water. The process is contained in a glove box (operated at negative pressure) to prevent tritium contamination to the environment. The feed is filtered to remove calcium carbonate, and the solid waste is mixed with the tritium from permeate into cement. If any tritium leaks into the glove box, a cryogenic cooler will condense the water from the air and return it to the cement mixer.[9]

A polyphosphazene membrane is used in separating tritium from water. Several membranes, configured into hollow cylinders, are placed into modules. This configuration offers a large surface area for separation in a small volume. The modules are mostly representative of one stage. The modules will operate much like heat exchangers and the streams are fed to each module shell side. The retentate also leaves shell side, while the permeate leaves tube side. Filters are used for removal of solid ( $CaCO_3$  particles) and the filtered water will be sent to the membrane. A cryogenic condenser will be used to condense the tritiated water vapor from the air. Liquid nitrogen is the most commonly used as a low-temperature refrigerant for the system. A glove box with air lock chambers and 10 sets of glove ports will encase the entire process. The concentrated tritiated water will be used to make the cement.

The author C.G. Woods claims that the separation factor is 2.33 which corresponds to 43% rejected tritium. 95.00% of the tritium is removed from the feed. Multiple stages would be required to obtain the desired separation.

#### E. Arsenic

Arsenic is toxic and dangerous arsenic concentrations in natural waters is now a worldwide problem. 21 countries in different parts of the world are affected by groundwater arsenic contamination. Many new technologies for arsenic removal have come up in the recent years.

1) *Adsorption and Precipitation*: The proposed technique of arsenic removal is that first pretreatment step is to oxidize arsenite to arsenate by potassium permanganate, second step is precipitation based on arsenic compound solubility with ferric sulfate and slaked lime under pH adjustment, and the last complementary step is followed by the adsorption of the bentonite which is enhanced by activated carbon and organic adsorbent. Experimental results show that under the optimal condition the removal efficiency of arsenic in the waste water is better than 99.99%, or the concentration of arsenic is from its original 100mg/l reduced to less than 10µg/l accordingly.[10]

The sample collected for experiment by Yue Li and Min Xi (2009), are analysed by the atomic adsorption spectrometer. Besides bentonite and activated carbon, an organic adsorbent is also used which mainly consists of vegetable protein.

The removal efficiency of arsenate is 30% higher than that of arsenite, hence  $\text{KMnO}_4$  has been selected to oxidize As(III) to As(V) at the beginning. Slaked lime ( $\text{Ca}(\text{OH})_2$ ) have been applied in pH adjustment and precipitation cooperated with ferric sulphate ( $\text{Fe}_2(\text{SO}_4)_3$ ), because of the high efficiency of the removal of arsenic based on the low solubility of  $\text{Ca}_3(\text{AsO}_4)_2 \cdot 3\text{H}_2\text{O}$  (solubility of 0.013),  $\text{Ca}_3(\text{AsO}_4)_2 \cdot \text{Ca}(\text{OH})_2$  (solubility of 0.0023-0.0048), and  $\text{FeAsO}_4 \cdot 2\text{H}_2\text{O}$  (insoluble)

Bentonite has been considered to complement removal of arsenic, which enhanced by activated carbon and organic adsorbent. Bentonite can reach its absorbing equilibrium quickly with good saturated adsorption amount, and the most important is that it is cost-efficient and easy to obtain. Activated carbon is a classical kind of adsorbent with better saturated adsorption amount, and it is used in a broad way presently. The arsenic in waste water can be reduced from 100mg/l to 13.7 µg/l under the optimal conditions.

Arsenic adsorption amount of bentonite is about 5.1mg/g. Similarly, using the same procedures the saturated arsenic adsorption amount of activated carbon (18.3mg/g) and organic adsorbent (21.2mg/g) are also obtained. This process is also efficient to remove gallium and indium in the waste water. After treating the concentrations of gallium and indium are from original 20 mg/l and 5 mg/l respectively reduced to lower than the detecting limit of AA analysis.

### III. CONCLUSION

The review was done on the purification of industrial waste water by studying the various methods of removing pyridine, Phenols, Phenoxides Chlorophenols, Tritium, Arsenic and Chromium. Various catalysts and unit operations were taken into consideration and were studied in depth to analyze the efficiency of the same.

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