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Removal of Phenol, Ammonia And Thiocyanate Either Alone or in Combination By the Adsorption with Steel Slag

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Abstract: *Wastewater any industry is of very complex nature as it contains multiple toxicants. The evaluation of individual toxicant in isolation and in the presence of other toxicants is necessary. Adsorption of phenol, ammonia and thiocyanateeither alone or in combination has been evaluated against the parameters affecting the adsorption. Dosage of adsorbent, initial concentration of the toxicant, time of adsorption process and pH of the solutions and studied with the help of fractional factorial Experimental Designs* 2^{4-1} *and FTIR analysis. It is found that removal of ammonia by the adsorption on the steel slag is favored under the presence of phenol. Similarly, adsorption of phenol and thiocyanate is also found increased in the presence of phenol.*

Keywords: *phenol, ammonia, thiocyanate, adsorption*

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I. INTRODUCTION

Phenol is found in petroleum refineries, petrochemical, steel mills, coke oven plants, coal gas, synthetic resins, pharmaceuticals, paints, plywood industries and mine discharge. Phenol causes many subtle effects to the biota, such as reduced fertility, decreased survival of the young, and inhibition of growth. High exposures to phenol may be fatal to human beings; infants appear to be hyper susceptible to phenol [1].However, adverse effect of the phenolic compounds is not seen on the aquatic organism [2][3].

Ammonium is always related to the eutrophication of surface water. Ammonium is also found in the waste effluents of industries such as coking, fertilizer plant and petroleum refining in the range of 5-1000 mg/L. Ammonium is also found in municipal waste in the range of 10-200 mg/L. Ammonia is known to be toxic to all vertebrates causing coma, death and neurological disorder [4].

Thiocyanate is generated from the coking of coal, quinine factory, base metal mills and gold mines. Salts of thiocyanate like potassium, ammonium, sodium are used as a herbicides. Thiocyanate is considered as less toxic however its effect the growth and reproductive system of the fish are widely reported [5]. Several industries convert its cyanide present in the effluent to thiocyanate. Thiocyanate is more stable than the cyanide which make its treatment difficult [6].Steel slag as an adsorbent is used in this studies. Steel slag is having commercial value and is known for its use in as aggregate in bituminous mixes, cement ingredient, concrete aggregate, antiskid aggregate, ceramic ingredient, railroad ballast, thermal insulator, landfill daily cover material, sand capping, obtaining calcium carbonate and also in use as a carbon sequestration[7].

Adsorption is still a major treatment method used for elimination of organic compounds from the wastewater. A rising trend is seen in patenting in the area of adsorption[8]. Adsorption technique is useful for the removal of phenol at high as well as medium level of concentration of influent [9]. The most usual adsorbents for water treatment are activated carbons (ACs)[10]. Amberlite, XAD-4, a polymeric adsorbent is used in several locations around the world to remove phenol from wastewater [11]. Absorption capacity of phenol can be improved by electroadsorption on activated carbons fiber[12]. Polymeric resins represent an alternative to activated carbons for adsorption of phenols. The resin's capacity for phenol increases with increasing phenol concentration. [10]. Olive mill waste [13][14], cereal by products[15], palm oil seed husks with sawdust and tannery leather scraps[16], manganese/silicon tailing waste [17], rice husk ash [18], cherry stone [19], Tobaco Residue [20], TyphaOrientalis Pres[21] are reduced to activated carbon for utilizing them as adsorbent. Zeolite [22], coking coal [23], acid treated slag [24], chemically modified bentonite[25], LuffaCylindrica Fiber[26] are also suggested as better adsorbent. Most of the studies concluded adsorption of phenol is better described by Langmuir and Freundlich isotherm and the Pseudo second order model.

Adsorption of ammonia by different adsorbent has been tried for several years. The use of waste material before its disposal was the essence of most of the researchers. Natural Australian zeolite [27], natural Chinese clinoptilolite[28], natural andNaCl-modified zeolites[29], Canadian zeolite [30], bentonite/chitosan beads[31], Canadian zeolite Composite having zeolite, limestone, activated carbon and rice husk carbon ordinary Portland cement (OPC) as a binder [32],Lime stone and granulated activated carbon[33],Strawberry leaves and stems, Boston ivy leaves and stems, southern magnolia leaves and poplar leaves[34],Ceramic made from Kanuma mud, Akad-ama mud, zeolite powder, soluble starch, and Na_2SO_4[35],Modified polyurethanes using alginate [36] are used for the adsorption of ammonia. Most of the studies used zeolite in natural form or its modified form for the adsorption of ammonia. The studies report that ammonium/ammonia adsorption follows the pseudo-second order model in most of the cases. Freundlich isotherm is found to have better correlation as compared to Langmuir model.

Literature shows that shows that Calcinedhydrotalcite[37], surfactant-modified coir pith at 2 pH [38], Fe(III)/Cr(III) hydroxide waste at 4 pH[39], Ferrihydrite[39],synthetic hydrotalcite sol[40],AgCl nanoparticles-loaded hydrotalcite[41] are used as adsorbent for the adsorption of thiocyanate. Studies show that adsorption follows Pseudo second order and Langmuir Model.Some studies show that presence of some toxicants does affect the adsorption of target toxicant. The presence of ammonia helps in increasing the adsorption of phenol on the resin [42].99 % phenol and 91% ammonia was removed by the powder activated carbon for treatment of cokeoven wastewater [43].The aim of the present study is to investigate the effect of phenol, ammonia and thiocyanate on adsorption either alone or in combination. Fractional Factorial Experimental Designs 2^{4-1} is used to reduce the required number of experiments and to obtain better response [44]. JMP software is used to find the correlation. Treated and untreated slag is characterized by IR studies.

II. MATERIALS AND METHODS

Steel slag is obtained from Steel Authority of India Limited (SAIL) located at Bokaro, India. The choice of this material is based on its low cost, considering its abundance. Steel slag of the desired size was obtained by sieving. The crushing was not required as the slag obtained had the particle size of desired range. The 150 microns retained size is selected for the purpose of experimentation. Size selected is washed with distilled water and dried in an oven at the temp of 150^oC for 24 hours, then kept in a desiccator for further use. The washing and drying step helped in removing the soluble and other impurities from the steel slag and making the adsorption sites available for adsorption on the steel slag.

All the necessary chemicals used in the study were of analytical grade. Phenol of AR grade of 99.5 % purity is obtained from CDH (P) LTD. 100 mg/L of $NH4^+N$ stock solution is prepared by dissolving 0.382g of NH_4Cl in distilled water. Standard Thiocyanate stock solution was prepared by dissolving 1 g of AR grade Potassium Thiocyanate (CDH (P) LTD) in 1000 ml of distilled water. Concentration of phenol is determined by the use of direct photometric D1783-13408. To determine the concentrations of $NH4^+N$ to phenate method is used [45]. Concentration of thiocyanate is determined by iron (III) and measuring the intense red color [46].The concentrations of phenol, ammonia and thiocyanate in samples are determined by UV spectrophotometer (Milton Roy Company (USA), range 340-960 nm). All experiments are performed in triplicate. The pH of the solution was measured with a Japsin (Japsin product,India) pH Meter. XRF is done by WDXRF Spectrometer - Bruker S4 PIONEER,USA. SEM images are obtained from Zeiss Model: V5:05 (SIGMA), Germany. XRD is done using Rigaku Smartlab Guidance CuKa irradiation (1.54 A^0, 40 kV, 30 mA),Japan. FTIR analysis is performed using Vertex 70, Bruker,USA.

Adsorption Studies

All experiments are done in the batch mode. Standard solutions of phenol, ammonia, thiocyanate, phenol-ammonia (1:1), phenol-thiocyanate (1:1), ammonia-thiocyanate (1:1) and phenol- ammonia-thiocyanate (1:1:1) are prepared.

Table 1: Experimental parameters

Expt No.	Dosage (gm)	Conc (Mg L^{-1})	Time (Min)	pH
1	1	1	120	12
2	16	16	120	12
3	1	1	5	2
4	16	16	5	2
5	1	16	5	12
6	16	1	5	12
7	16	1	120	2
8	1	16	120	2
9	8.5	8.5	62.5	7

As shown in Table 1Fractional Factorial Experimental Designs 2^{4-1} is used to reduce the required number of experiments. The parameter taken for the studies are dosage of steel slag, pH of the solution, time for adsorption and Initial concentration of the solution. The level of each parameters is taken after due consideration of studies done by other researchers.The amount of toxicant adsorbed per unit mass of the adsorbent and percentage removal was evaluated by using the following equation [47][48].

$$\% \text{ removal} = ((Co-Ct)/ Co)*100 \qquad (1)$$

Where, Co is the initial concentration of phenol, Ct is final concentration in mg/L at any time t, V is volume of solution in mL of adsorbate solution and m is the dose of slag in gram.

III. RESULTS AND DISCUSSION

Characteristics of Steel slag

The steel slag is a whitish dusty and the size of the grain varies from .75 microns to 4 mm in diameter. Sieve analysis is done and it shows that the steel slag is a well graded. The steelmaking slag is characterized by sieve analysis,Table 2 shows the slag composition done by WDXRF Spectrometer - Bruker S4 PIONEER. Figure 1 shows the SEM images obtained from Zeiss Model: V5:05 (SIGMA) shows the porous nature of the steel slag which indicates that abundance of adsorption sites on its surface. The elements like O, Si, Ca, Al, Mg, C and K are found to be present in the steel slag. The surface morphology of steel slag particle shows sharp edges, partly dense, and partly porous. The BET surface area, pore diameter and the pore volume measured for the sample is 11.6 m^2/g, 4.06 nm and 0.010 cc/g respectively. According to the IUPAC, the material can be classified as mesoporous material. This is also confirmed by the studies done elsewhere [49].

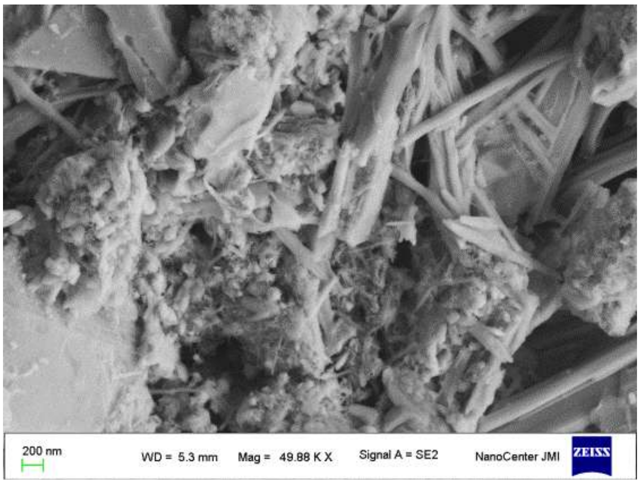

Figure 1: SEM of Steel Slag

Table 2: XRF of steel slag

Sample	Slag
CaO	46.21%
Fe_2O_3	14.89%
SiO_2	9.52%
MgO	2.51%
Al_2O_3	1.94%
P_2O_5	1.20%
TiO_2	0.56%
MnO	0.52%
Cr	0.07%
K_2O	0.02%
Na_2O	0.04%
Cl	0.01%
Cu, PPM	265.55
Nb,PPM	42.59
S, PPM	4835.20
Sr, PPM	201.72
V, PPM	370.60
Zr, PPM	13.02

Table 1 shows different experimental conditions like dosage of steel slag, Initial concentration of chemical, Time given for the adsorption and the pH condition which is applied. Table 3 shows the percentage removal of Phenol, Ammonia,Thiocyanate, phenol in Phenol-Ammonia (1:1) Solution (PA), Ammonia is Phenol-Ammonia (1:1) Solution (PA) , phenol in Phenol- Thiocyanate (1:1) Solution (PT), Thiocyanate in Phenol- Thiocyanate (1:1) Solution (PT), Ammonia in Ammonia- Thiocyanate (1:1) Solution (AT), Thiocyanate in Ammonia- Thiocyanate (1:1) Solution (AT), phenol in Phenol-Ammonia- Thiocyanate (1:1:1) Solution (PAT), Ammonia in Phenol-Ammonia- Thiocyanate (1:1:1) Solution (PAT) and Thiocyanate in Phenol-Ammonia- Thiocyanate (1:1:1) Solution (PAT).

Table 3: Response to experimental parameters

Expt				PA		PT		AT		PAT		
No.	P	A	T	PPA	APA	PPT	TPT	AAT	TAT	PPAT	APAT	TPAT
1	25.3	63	29.3	30.7	65	24	20.1	51.2	35.3	28.5	68.53	27.8
2	98	31	20.7	97	33.4	92	17.3	25	27.8	98.3	35.7	18.3
3	60	45	47	62.3	47.87	57	31.1	41	62.1	58.3	49.3	10.3
4	50	50.7	15.4	53.7	53.7	44	10.3	45.2	30.2	45.3	42.1	20.87
5	55	20	15	57.3	27.2	45	7	17	35.6	49	15	10.3
6	43	65	30.2	49.3	66.2	37	15.1	60	40.2	37.3	60.2	25.4
7	95	72	59.1	97	75	88	42	55.5	70.3	83	63.7	47.2
8	20	25	22	30	28.3	40	10	22	30.1	15.7	22.1	15.1
9	40	40	35	45.9	42.1	30	21	33.2	41.1	35.2	32.2	29.3

Ammonia Removal

Figure 2 shows that removal of ammonia by the adsoprtion on the steel slag is favored under the presence of phenol. Whereas ammonia removal is found to be reduced in the presence of Thiocyanate. While, the presence of phenol marginally improves the adsorption of ammonia on to the steel slag.

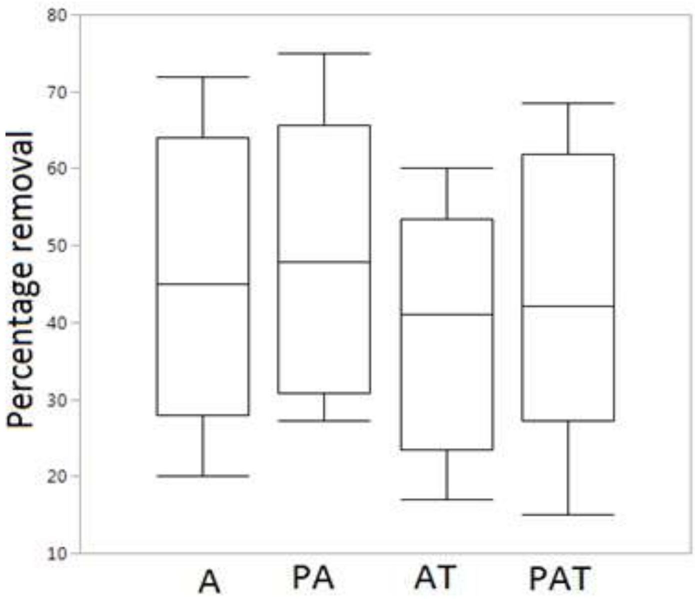

Figure 2: Percentage ammonia removal

As can be seen in Figure 3Increase in dosage of slag has shown the increase in adsorption of ammonia. Maximum of 75 % removal of ammonia is possible in the presence of phenol. Increase in initial concentration of the solution has the negative impact on the adsorption of the ammonia. Increase in time period of adsorption helped in improving the adsorption of ammonia however, no effect of increase in time is found on the adsorption of ammonia in AT solution. Increase in adsoption of ammonia is found under acidic conditions in PA and A, whereas increase in adsorption of ammonia is found under alkaline condition in PT and PAT solutions.However, overall removal of ammonia from PT aand PAT solution remains low. Therefore, it can be said that, ammonia adsorption is favored in presence of phenol, with high dosage of steel slag, low intial concentration of solution and under acidic conditions[43].

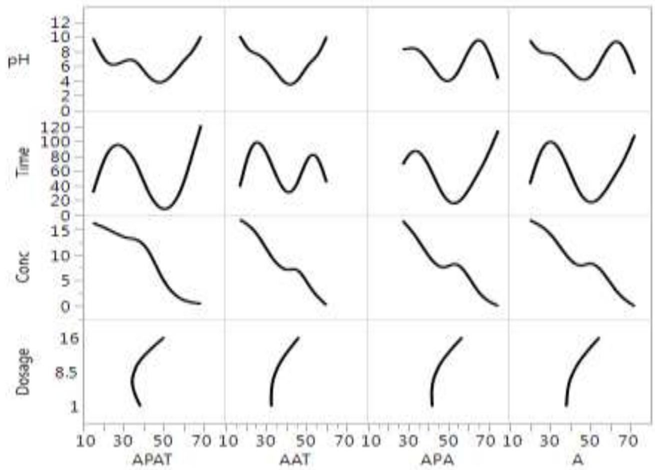

Figure 3: Ammonia removal under different parameters

Phenol Removal

As described in the ammonia adsorption that presence of phenol favors the adsoption process similarly, the adsorption of phenol is also found to be favorable in presence of ammonia as shown in Figure 4. Presence of thiocyanate decreases the adsorption of phenol on the steel slag. However, increases percentage adsorption is can be seen as the dosage of the steel slag is increased in the process of adsorption. Figure 5 shows that increase in initial concentration of the solution, time of contact as well as increase in pH are found to be favoable in the adsorption of the phenol on to the steel slag.Maximum removal is found to be 57.3 % in the presence of ammonia.

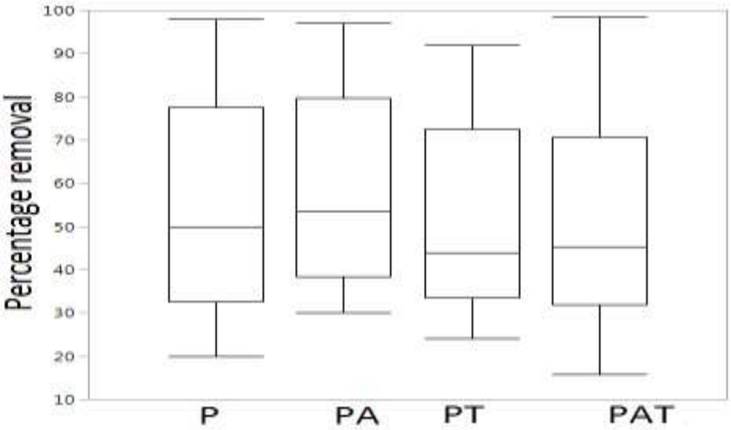

Figure 4: Percentage phenol removal

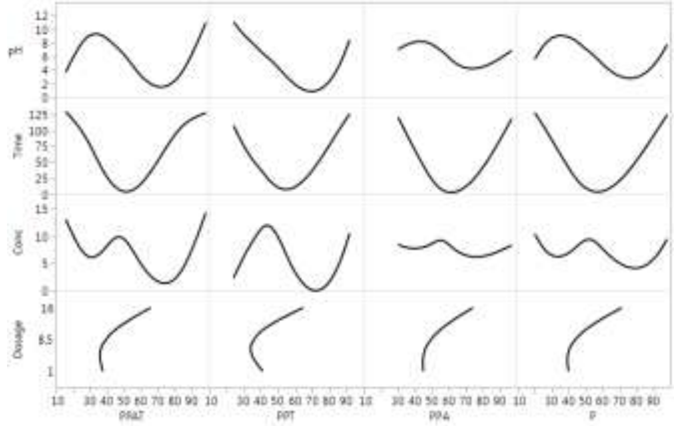

Figure 5: Phenol removal under different parameters

Thiocyante removal

Analysis of removal of thiocyanate under different solution is shown in Figure 6 and Figure 7. Thiocyanate removal reached to its maxium at 70.3 % in the presence of ammonia. However, in presence of both phenol and ammonia decrease in removal of thiocyanate has been observed. Increase in dosage of steel slag has no significant improvement in the adsorption of the thiocyanate. Thiocyanate removal is found to be improved at lower initial concentration of the solution, increase in time of contact and under acidic conditions.

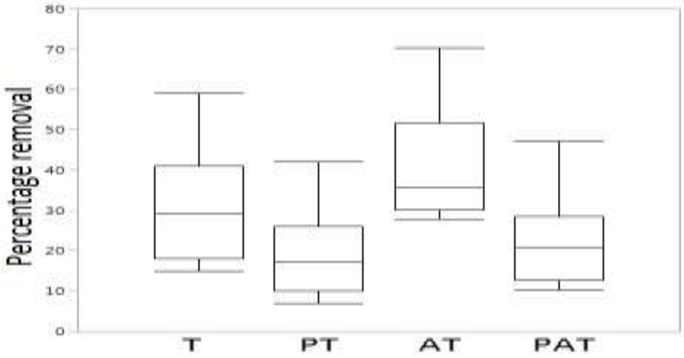

Figure 6: Percentage Thiocyanate removal

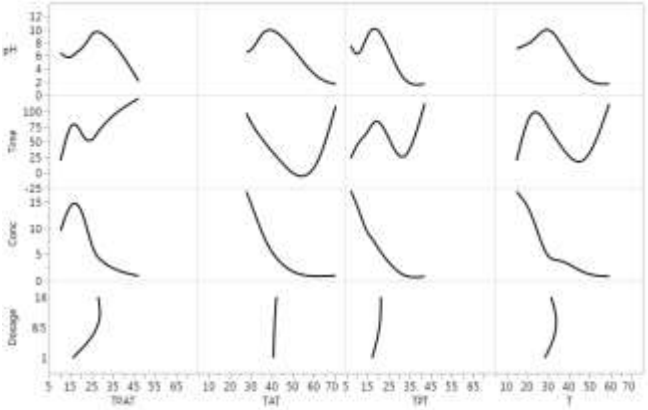

Figure 7: Thiocyanate removal under different parameters

IV. Correlation

Correlation between the measuredtoxicants in the solution with parameters in

Table **4** shows that increase in dosage has the positive effect on the percentage adsorption in all types of solutions. Increase in initial concentration has the negative impact on the all the solutions where ammonia and thiocyanate were present. However, increase in initial concentration did not show negative effect on the adsorption.Increase in time has shown increase in percentage of adsorption in all types of solutions except for adsorption of phenol in PA, Thiocyanate in AT and Ammonia in PAT solution.Increase in pH has negative effect on the adsorption of ammonia and thiocyanate in AT solution. Adsoprtion of phenol is also affected at higher pH in the presence of ammonia.

Table 4: Correlation of Solutions with parameters

Solution Mix	**Concentration measured**	**Dosage**	**Conc**	**Time**	**pH**
P	P	0.3303	0.0349	0.8149	-0.0628
A	A	0.0699	-0.6902	0.0594	0.0976
T	T	0.1023	-0.7821	0.1987	-0.4084
PA	P	0.2148	0.1960	-0.9089	-0.7734
	A	0.1667	-0.4090	0.1904	0.7028
PT	P	0.3530	-0.0447	0.6111	0.1528
	T	0.0251	-0.7183	0.1817	0.2871
AT	A	0.3129	-0.7604	0.1448	-0.3482
	T	0.0613	-0.7751	-0.0540	-0.4750
PAT	P	0.5702	0.0123	0.1764	0.0615
	A	0.1312	-0.8295	-0.0724	0.2962
	T	0.4380	-0.4107	0.3519	0.0296

TIR analysis

The chemical structure of the adsorbent can provide the vital information on the functional groups participating in the adsorption process [50]. FTIR analysis is performed on the slag before and after the adsorption of the phenol, ammonia, and thiocyanate either alone or in combination as described above.KBr pallets were made for the analysis. The results are shown inFigure 8 and Figure 9. Change in the vibrations throughout the spectrum suggest that the complex nature of the sample [51]. The periclase (MgO),Si-O-Si and Al-O-Si is found between 800 to 1114 cm^{-1}[52][53]. The oxides of Fe are found at 476 and 786 mm^{-1}[54]. It is observed in the FTIR of the steel slag after adsorption of phenol, that aromatic C-H bending exists in 680 to 860 cm^{-1} and 1585 to 1600 cm^{-1}. In particular stretching for phenol is found is the region of 507 to 1007 cm^{-1} [55].The vibrational changes of high intensity is observed from 405 to 786 cm^{-1}. Most of the peaks can be seen vanished in this region after adsorption. This shows that this region is mostly contributing towards the process of adsorption. Medium intensity changes are observed in 1560 to 1676, 3176, 3178, 3197 to 3411, 3335 and 3537 cm-^{1}. The adsorption of phenol on the slag is the direct effect of the participation of silicates, aluminates and periclase present in the sample.

While studying the effect on the surface of the steel slag after the adsorption of ammonia it is found that, the change in the band is observed from 432-555, 671-675, 705-709, 806-1286, 1411-1517, 1728- 2790, 3656-3990 wavelength. Change in band is also found in the region of 1475 to 1650 which belongs to $–NH_2$, -NH and NH_3 bands. Comparing the spectrum of steel slag after the ammonium adsorption with the spectrum before adsorption confirms the ammonium being adsorbed on the steel slag. Participation of Si-O-Si in the process of adsorption cannot be ignored.

Vibrational change is observed for thiocyanate in the range of 2125 to 2250 mm^{-1} for the sample after adsorption with the thiocyanate [56]. Vibrational changes of high intensity are observed 405 to 1250 which shows participation of oxides of magnesium and iron participating in the process of adsorption. Medium intensity changes are observed in 2500 to 3000 mm^{-1}. FTIR studies confirm the active role and availability of the functional groups in the process of adsorption [57]. Patterns of PA,PT,AT as well as PAT also shows the vibrations changes in the regions as described above.

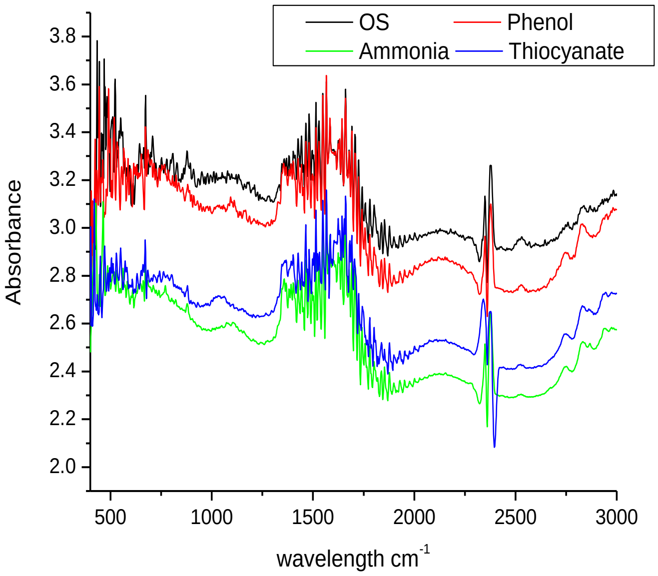

Figure 8: FTIR of steel slag under after and before adsorption

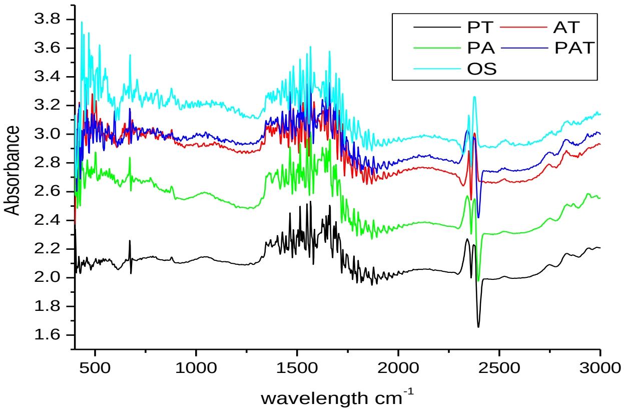

Figure 9: FTIR of steel slag under after and before adsorption under mixed conditions

V. Conclusion

Adsorption of phenol, ammonia and thiocyanate either alone or in combination is studied in details with the parameters affecting its adsorption capacity. It is found that removal of ammonia by the adsorption on the steel slag is favored under the presence of phenol. Similarly, adsorption of phenol and thiocyanate is also found increased in the presence of phenol. Maximum of 75 % removal of ammonia is possible in the presence of phenol. Maximum phenol removal is found to be 57.3 % in the presence of ammonia. Thiocyanate removal reached to its maxium at 70.3 % in the presence of ammonia. Thiocyanate is found to affect the adsorption of phenol as well as ammonia. Ammonia removal is found to be reduced in the presence of Thiocyanate. Ammonia adsorption is favored in presence of phenol, with high dosage of steel slag, low intial concentration of solution and under acidic conditions. Increase in initial concentration of the solution, time of contact as well as increase in pH are found to be favoable in the adsorption of the phenol on to the steel slag. Thiocyanate removal is found to be improved at lower initial concentration of the solution, increase in time of contact and under acidic conditions. Overall the coexistance of phenol, ammonia and thiocyanate would lower the perecentage adsorption of all toxicants under consideration.

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