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Potential of Hydroxamic Acid in Determination of Phenol in Industrial Waste Water

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A spectrophotometric method based on N-Phenyl benzo hydroxamic acid reaction is described for determination of phenol . Phenol reacts with hydroxamic acid in acidic media with V (V) to give purple complex . It has maximum absorbance of 522 nm . Beer's law is obeyed in the range 0.006 to 0.03µg ml^{-1}.The reaction conditions and other analytical parameters have been optimised . The above method is found to be highly selective and is characterized by its simplicity with accuracy and precision .It has been successfully applied to determine phenol in industrial waste water sample of six areas of Mandideep , Bhopal.
Key Words : Phenol, N- Phenyl benzo hydroxamic acid, spectrophotometric, industrial waste water.

Introduction : Phenol is toxic substance found regularly in environment. It is introduced into the environment through industrial discharges from petroleum refineries [1-2] , drug manufacturing units[3-4] , antioxidants[5-7] ,oil refineries[8], coke processing waste water[9] and coke oven plant[10]. Phenol traces are also found in tobacco smoke[11] ,cigarette smoke[12], wine[13] ,drugs[14] and automobile exhaust [15] . Phenol is also obtained from catabolism of natural or chemical products , eg.- pesticides . Phenol is harmful to personal health as exposure to high concentration of phenol in air causes paralysis and severe injury to various important body organs . The US environmental protection agency recommends the limit of 1 µ/litre of phenol/litre in water used for drinking purpose .

Number of analytical methods are proposed for determination of phenol. spectrophotometric analysis[16-19] involving reagent like p-amino phenol [20], 4- aminoantipyrine[21], N-diphenyl benzomidine[22] , sodium nitro prusside[23]are reported. Chemical analysis , liquid chromatographic analysis[24] , HPLC analysis [25-27], flow injection analysis , electrochemical analysis are some other methods.

This study demonstrates a spectrophotometric method for determination of phenol by using versatile metal extractant N-phenyl benzo hydroxamic acid. Determination conditions were optimized in the course of analysis . This proposed method was found to be highy selective towards the determination of phenol. It has been applied for determination of phenol in waste water.

Experimental

Instruments : "SYSTRONICS SPECTROPHOTOMTER 1700" model was used for electronic spectral measurements with 10 mm matched quartz cells. A Hanna 8521 model pH meter was used for pH measurements.

Reagents : All the chemicals used were of AR grade. Double distilled water was used throughout the experiments.

Material and method : All chemical were of analytical reagent grade. All standard and sample solution were made up with distilled water .

Stock phenol solution – 1 mg mL^{-1} stock solution of phenol was prepared in distilled water . Working standared were prepared by the appropriate dilution of stock.

N – Phenyl benzo hydroxamic acid was prepared according to the method given by Priyadarshini and Tandon [28] and solution was prepared in chloroform.

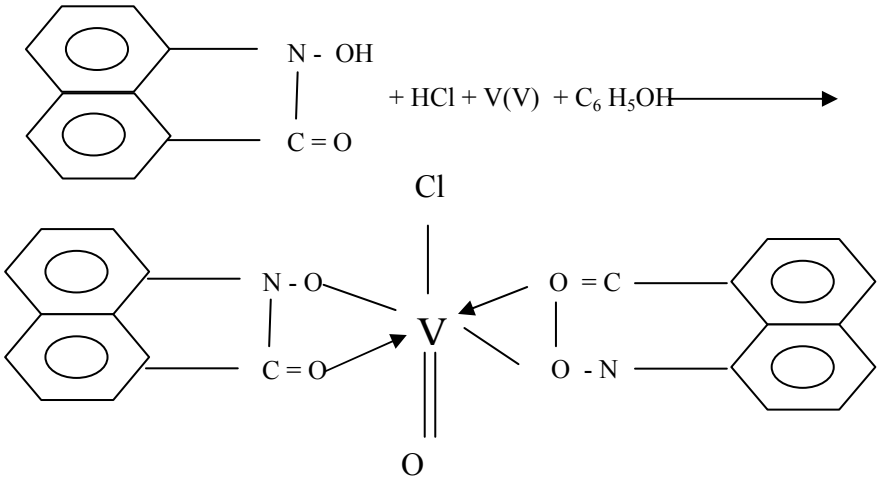
Ammonium meta vanadate solution - saturated solution was prepared by dissolving in distilled water.

Hydrochloric acid solution – 4M HCl solution was used to provide acidic medium .

Procedure : To aliquates of working standards containing µg of phenol , 1ml of N-PBHA solution in chloroform was added .This was followed by addition of 1 ml V(V) solution and 1 ml 4M HCl solution . The purple colour appeared in chloroform layer which was separated and diluted to 25ml. This coloured dye has maximum absorbance at 522 nm.

Result and Discussion :

Expected Reaction :



(Purple dye)

Beer's law was obeyed in the range of 0.006 to 0.03 µg ml^{-1} . The absorption spectra of the coloured product formed in proposed method show maximum absorbance at 522 nm. The calibration curve is shown in figure 1.

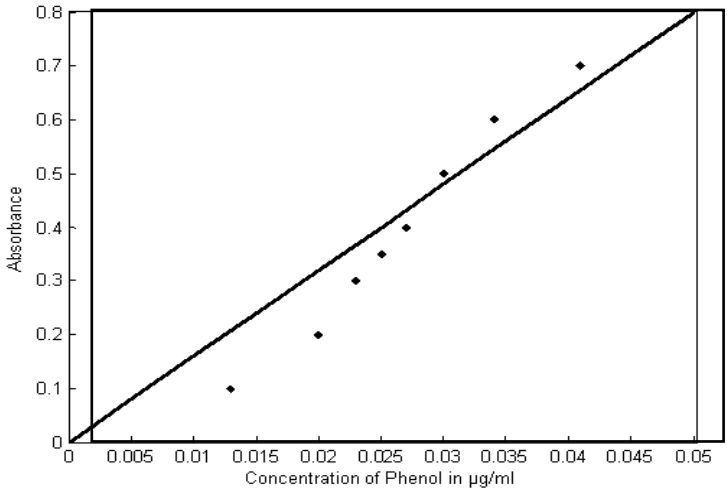


Fig.1: Calibration curve for the determination of phenol

Effect of reagent concentration – Best results were obtained by using 1ml of N-phenyl benzo hydroxamic acid solution in chloroform . There was no change at higher concentration, where as at lower concentration there was decrease in absorbance values . This is clear in figure 2.

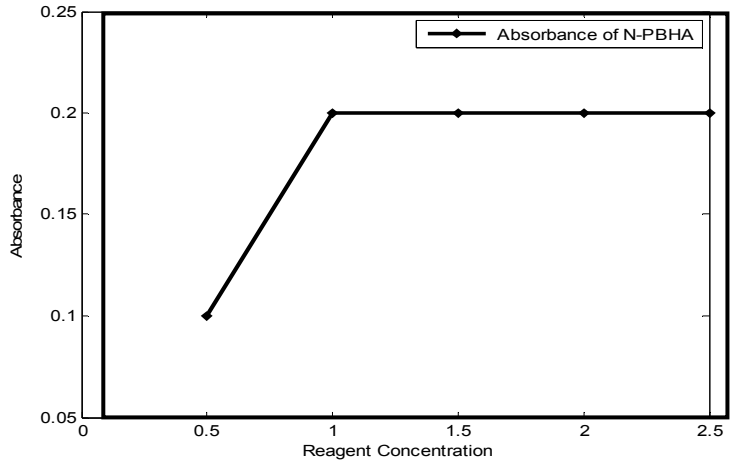


Fig.2 : Effect of concentration of N-Phenyl benzohydroxamic acid

Effect of time and temprature - Room temperature was found to be most suitable for obtaining maximum absorbance. Above and below this , absorbance was markedly affected. Maximum required time for colour development was found to be 10 min. The colour obtained remained stable for several days .

Effect of molarity of acid – Most effective result were obtained by using 4M . Increase or decrease in molarity results in fading of colour. This is shown in figure 3.

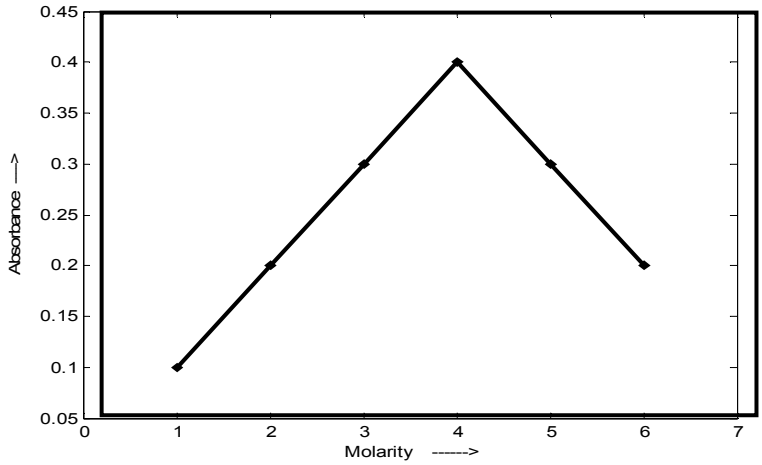


Fig. 3 : Effect of molarity of acid

Order of addition of reagent – The order followed was : Phenol solution, PBHA solution , V(V) saturated solution , HCl solution followed by distilled water . If the order is changed sensitivity decreases.

Effect of foreign species – To check the validity of the method, effects of several species commonly found along with phenol, on the reaction were studied. This was done by addition of known amount of these species to 3 µg phenol prior to its analysis by the proposed method. The tolerance limits for various interfering species are shown in table :1.

Table 1 : Effect of foreign species

Foreign Species	Tolerance level ($\mu g\ mL^{-1}$)
Methenol, Ethenol , Benzene	980
Toluene	290
Formaldehyde, aniline	280
o-nitrophenol	60
o-cresol, p-cresol, m-cresol	750
Hg^{2+}, Ca^{2+},Pb^{2+}	800
Al^{3+},Fe^{3+}	1150
PO_4^{3-},SO_4^{2-} ,CH_3COO^-	1350
S^{2-}	90

Table 2 : Comparision with other spectrophotometric methods.

Method	_max (nm)	Beer's law range ($\mu g\ mL^{-1}$)	Remarks
4- amino antipyrene [17]	510	0.2 – 2	Metal ion interfere, long
p-nitroaniline [29]	530	0.1 – 0.4	Reagent is carcinogenic
p – anisidine, NCS [30]	650	0.05 – 0.4	Colored reagent blank, higher temperature required
Sodium nitropruside, hydroxylamine hydrochloride [19]	700	0.05 – 5.0	Interference of metal ions, less sensitive
NCS – Ammonia [31]	670	0.3 – 2.04	Less sensitive, metal ion interfere
Vanadium , N-hydroxyl-N,N'diphenylbenzamidine [18]	600	0.03	Extractive method
NBT, LCV[20]	595	0.02 – 0.22	Less sensitive, Non extractive, less time taking, non toxic reagent used, higher colour stability
Vanadium, N – Phenyl benzo hydroxamic acid (proposed method)	522	0.006 – 0.03	More sensitive , non extractive, quick, colour stable for several days.

Application : To check the validity of the method it was applied for determination of the phenol in industrial water of Mandideep area of Bhopal .

Table 3 : Determination of Phenol in water sample

Samples	Amount of phenol found in ($\mu g\ mL^{-1}$)	
		Proposed Method
Polluted waste water	W 1 Satlapur	0.75
	W 2 Khanpura	1.00
	W 3 Moizpura	1.01
	W 4 sarakia	2.08
	W 5 Jhalarkalan	3.11
	W 6 Nayapura	3.71

Conclusion : The proposed method is sensitive and the colour developed is stable for several days . This method can be compared favourably with other reported method in table 2 . This method has been successfully applied for the determination of phenol in industrial waste water of Mandideep .

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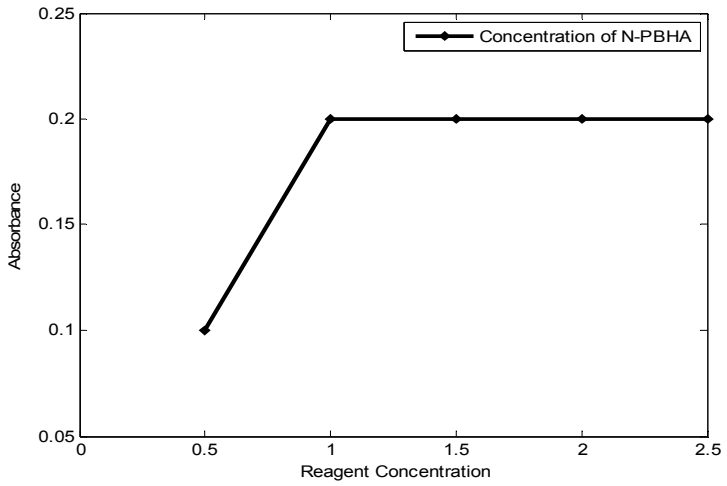
Concentration of N-PBHA
Absorbance
Reagent Concentration
0.25
0.2
0.15
0.1
0.05
0
0.5
1
1.5
2
2.5