

Electrochemical Degradation of indigo carmine Dye at Ru/graphite Modified Electrode in Aqueous Solution

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Abstract: The electrochemical degradation of wastewater containing indigo carmine dye was investigated by synthesized Ru/ graphite modified electrode. The kinetics of degradation by graphite and Ru/graphite modified electrode are compared. Indigo carmine is one of the most important textile dye. The wastewater of indigo carmine can be oxidized to Isatine, which is hydrolyzed to antranilic acid, a compound that is toxic to aquatic life. IR and SEM were observed for synthesized Ru/ graphite modified electrode. The thin film formation of Ru or encapsulated in graphite rod is observed from SEM/EDAX. The effect of concentration of the dye and the current on the kinetics of degradation were studied. The ICE values of different experimental conditions are evaluated. The anodic oxidation by Ru/ graphite modified electrode showed the complete degradation of water containing indigo carmine, which is confirmed by uv-visible and COD measurements. The dye is converted into CO₂, H₂O and simpler inorganic salts. The results observed for reuse of modified electrodes indicates that the Ru/graphite modified electrode would be promising anode for electrochemical degradation of indigo carmine. This method can be applied for the remediation of waste water containing organics, cost effective and simple.

Keywords: Indigocarmine, anodic oxidation, Ruthenium graphite modified electrode, Mineralization

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I. Introduction

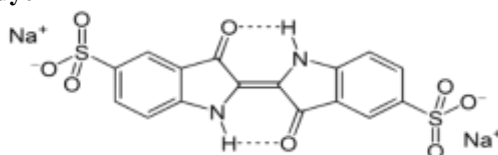
The textile industry consumes enormous amounts of water during dyeing and finishing operations. Typical medium-scale textile factories produce approximately 1000 m³ of wastewater per day. It is estimated that around 30% of the applied dyes remain unfixed and are discharged in the effluent. Dye bearing wastewaters are toxic for the environment since dyes are stable compounds and may cause carcinogenesis. Conventional biological treatment processes are often less successful because most dyes are barely or non-biodegradable. Indigo Carmine (5,5'-indigo disulfonic acid disodium salt) is a dark blue dye mainly used in the textile industry for the dyeing of polyester fibers and denim (blue jeans). Indigo Carmine is also used as dye in food and cosmetics industry. Several processes have been suggested for removal of Indigo Carmine and other dyes from wastewater including adsorption, photochemical and electrochemical methods [1]. The dye in the water-soluble system can be used as an industrial precursor material in the chemical process. Its characteristics are more readily soluble in aqueous solution. For example, using sulfuric acid to generate indigo carmine (Acid Blue 74,) it is a kind of common dye that may also be used as food additives or as a microscopic tracer in biology [2]. In recent years, the electrochemical technique has gained wide acceptance as a powerful alternative for wastewater remediation. It currently offers promising approaches for the prevention of pollution problems from industrial effluents. The application of electrochemistry to environmental pollution abatement has been the topic of several books and reviews. The strategies of electrochemistry include both the treatment of effluents and waste and the development of new processes or products with less harmful effects, often denoted as process-integrated environmental protection. Electrochemical oxidation or electro-oxidation (EO) is the most popular electrochemical procedure for removing organic pollutants from wastewaters. This technique has been recently used for decolorizing and degrading dyes from aqueous solutions [3]. The removal of indigo carmine from water and wastewater is a need of the highest order. Various technologies have been employed to remove IC from water and wastewater [4]. Electrochemical degradation of organic pollutants has been investigated by many researchers. And it has been proved as a cost-effective technology. Thus electrochemical degradation is a good choice of removing indigocarmine dye [5]. Large amounts of different dyes used in different industries. To avoid accumulation of dyes in the environment, powerful oxidation techniques are developed for the removal of dye in the industrial waste water [6, 7]. Wastewater from textile industries frequently contains significant amounts of non-biodegradable dyes [8]. Most of these dyes are toxic and potentially carcinogenic in nature and their removal from the industrial effluents is a major environmental concern [9]. A variety of hazardous pollutants are discharged into the aquatic bodies from several industrial streams [10]. Conventional methods for the abatement of water pollution from dye and textile industries like adsorption, absorption, incineration and biodegradation were found to be ineffective and expensive. Moreover, they pose secondary disposal problems

and associated increase in time and cost of operation [11,15]. Studies have shown that indigo carmine reacts with chemical oxidants such as, Peroxo disulphite ion, chloramine-T and ozone [16, 17]. In the recent years, environmentally friendly electrochemical methods are developed to degrade organic pollutants in waste waters. Anodic oxidation and electro-Fenton are the most usual techniques, since they have high degradation efficiency due to electrochemical reaction of OH radical as oxidant [18, 19]. However a limited number of papers have been published for the destruction of dyes by electrochemical methods [20]. In recent years, applications of chemically modified electrodes (CME) have made great success in various areas of research and development, such as material corrosion and inhibition, electro catalysis, electronics, biosensors and electroanalysis. The graphite electrode surface was mechanically polished using polishing sandpaper. After polishing, the electrode was rinsed with de-ionized water and left in the atmosphere for drying. The surface of the graphite electrode was renewed for each run [21].

II. Experimental

A solution of indigo carmine (IC) (E-Merck) was prepared [0.0001M]. The 8cm length and 0.63cm diameter graphite electrode (ALFA AESAR) was using. The 2.5cm length graphite electrode is dipped into the above solution. Prior to experiment the surface pretreatment of graphite electrode was performed by hand polishing of the electrode surface with successive grades of emery papers down to 3000 grit up to a mirror finish. The polished electrode was then degreased with 1:1 HCL solution, acetone and washed with running doubly distilled water and utilized for further electro deposition. The experimental set up is shown in the figure (1). Ruthenium was deposited on Graphite electrode from $RuCl_3$ (Arora matthey) solution. All chemicals used were of accepted grades of purity. The experimental apparatus is shown in fig (1). It consists of reaction chamber and a voltage power supply. The electrode system consists of a Graphite modified Electrode as anode and Pt electrode as cathode in case of degradation of dye by anodic oxidation with Graphite electrode. The distance of the anode and cathode was 2cm. The kinetic runs were carried out with Graphite electrode alone as anode. In another case, Ru (111) thin film deposited on a conductive Graphite electrode has taken as anode and Pt electrode as cathode. The experiment was run from 6 to 660 minutes with continuous stirring. The kinetic runs were carried out for different concentration of indigo carmine with different applied current. To account for the mineralization of the dye solution COD was measured at regular intervals of time. The pH of the solution before and after electrolysis was measured. A positive voltage was applied by using battery eliminator (NEULITE INDIA) and current output of 2.1mA-6.1mA using rheostat (INSIF INDIA). The decolorization and disappearance of indigo carmine was followed by using spectrophotometer (ELICO SL 171).

Properties of Indigo Carmine dye



Molecular formula: $C_{16}H_8N_2Na_2O_8S_2$

Molar mass: 466.36 g/mol

C.I.Number: C.I. 73015

Absorption maximum (water): 608 - 612 nm

CAS number: 860-22-0

Bulk density: 700 - 900 kg/m³



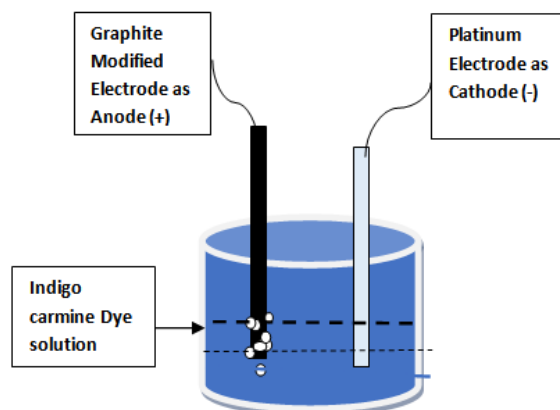


Fig. 1: Experimental set up for electrochemical degradation of IC Dye

III. Results And Discussion

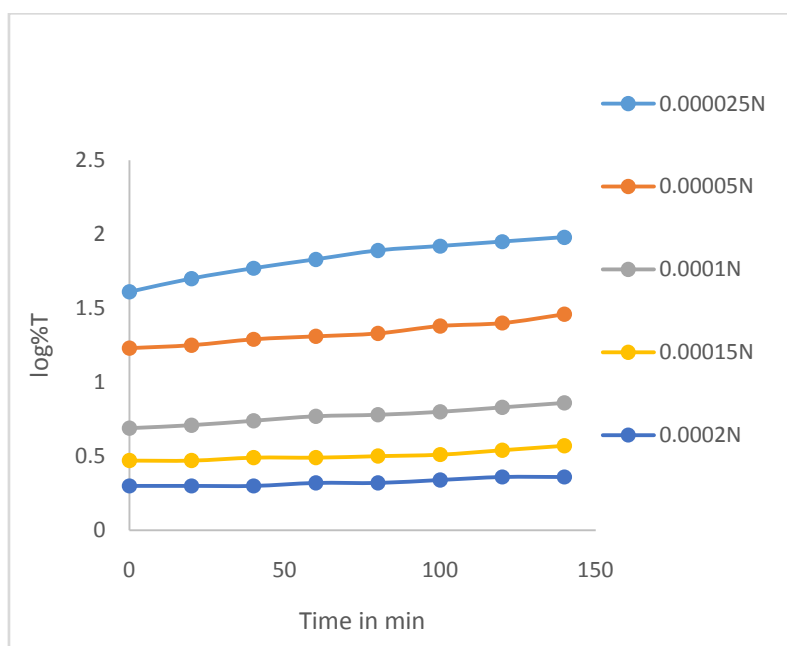
3.1 Reaction with Graphite as anode electrode

3.1.1 Effect of Indigo carmine

The reaction performed in the presence of IC (E-Merck) was prepared [0.0001M] with constant current (4.1mA). The change in concentration of the indigo carmine was recorded by change in color using spectrophotometer. A plot of log%T (percentage transmittance of light) versus time was linear up to 60% of the reaction indicating disappearance of IC follows first order kinetics. The rate constant values are given in table (1), the reaction rate decreased with increase in [IC]. Surface of the graphite electrode, decreases the rate of flow of current across the electrode-solution interface, which decreases the concentration of OH radicals. The pH value before and after the electrolysis remain constant. The COD for Indigocarmine solution before and after electrolysis were measured. (Table-1, Fig-2 and 3)

Table 1: Effect of [IC] on the rate of degradation and COD values

$10^4[IC]$	$10^4 k$ Sec ⁻¹	Effect of pH		COD Values in mg/l	
		Before degradation	After degradation	Before degradation	After degradation
0.25	0.99	6.23	6.27	464	16
0.5	0.61	6.54	6.59	512	16
1.0	0.46	6.96	6.99	624	32
1.5	0.26	7.13	7.15	816	48
2.0	0.19	7.32	7.36	992	32



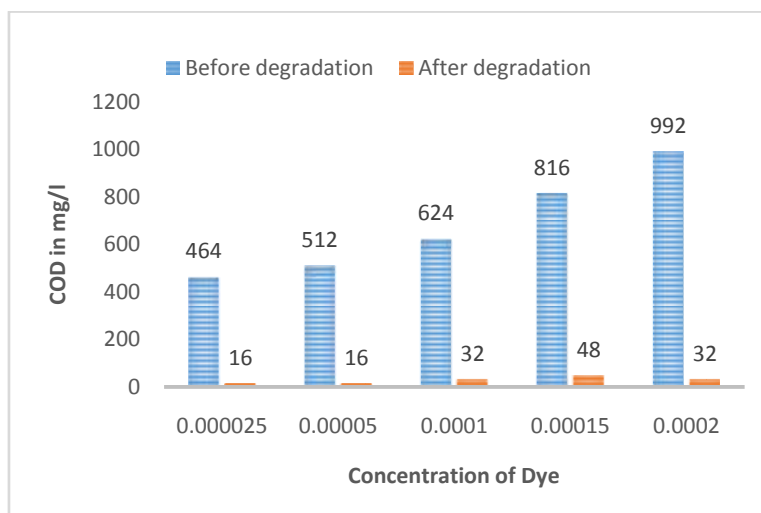


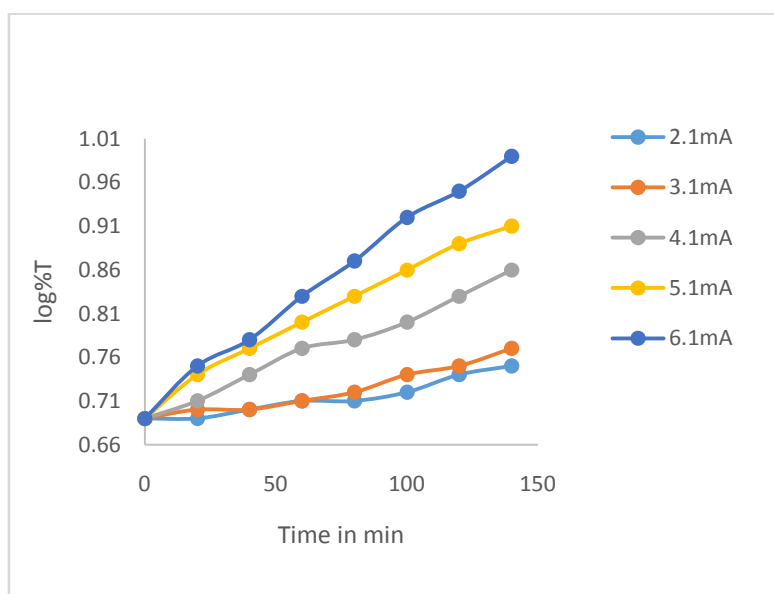
Figure 2 and 3: Effect of Concentration of IC on rate of degradation and COD Value

3.1.2 Effect of current on the rate

At fixed [IC] the rate of reaction increased with increase in applied current. The current is varied from 2.1mA-6.1mA. The increase of current increases the concentration of oxidizing intermediates and OH radicals, which increases the rate of degradation. The COD for different current of Indigocarmine solution before and after electrolysis were measured. (Table-2, Fig-4 and 5)

Table2: Effect of current on the rate of degradation and COD values

Current in mA	$10^4 k$ Sec ⁻¹	COD Values in mg/l	
		Before degradation	After degradation
2.1	0.15	624	48
3.1	0.23	624	16
4.1	0.46	624	32
5.1	0.57	624	32
6.1	0.80	624	16



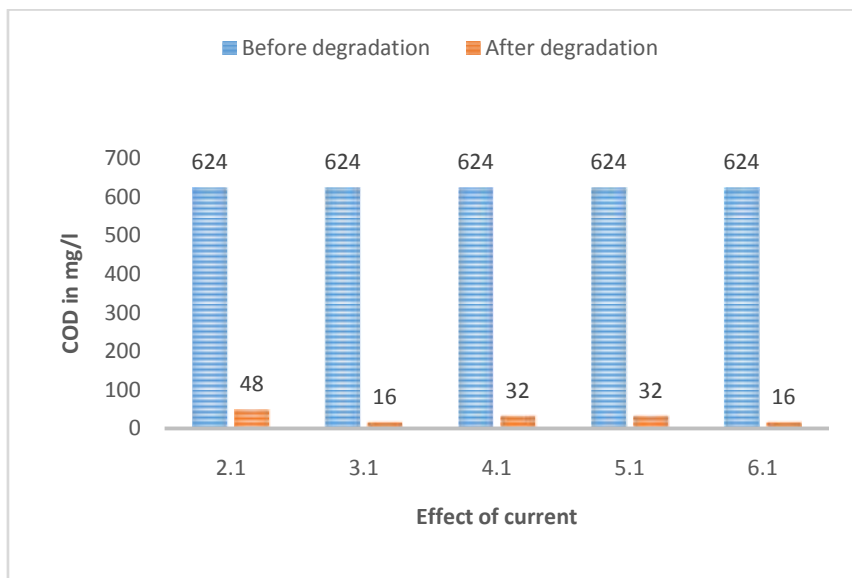


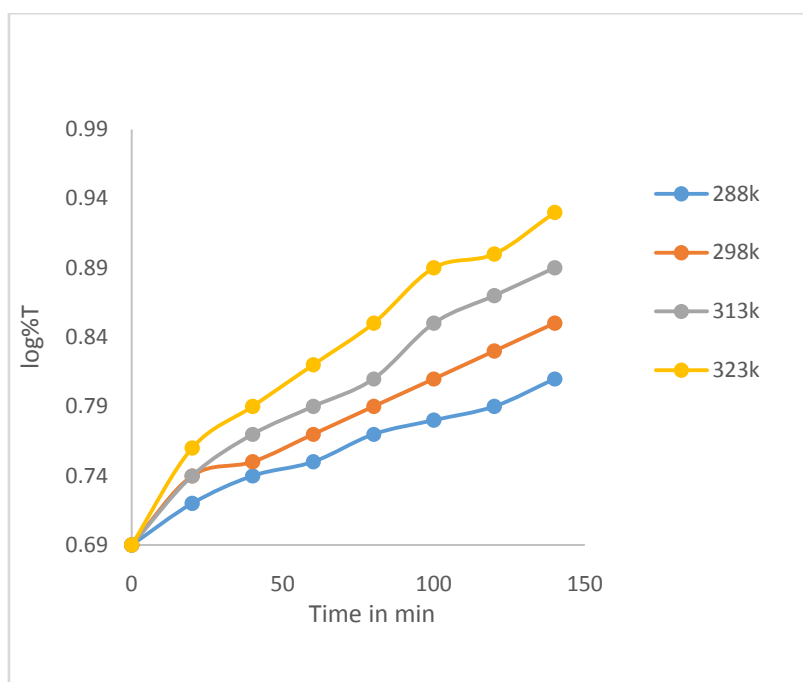
Figure 4 and 5: Effect of current of IC on the rate of degradation and COD Values

3.1.3 Effect of temperature

It is clear that an increase in the reaction temperature increases the dye degradation. It is observed that the rate of removal of color is not very significant at low temperature. However, the reaction is more significantly influenced at high temperature (Table 3). Since the diffusion rate is increased with temperature, an increase of temperature could bring about an increase in the degradation rate.

Table 3: Effect of Temperature on the rate of degradation and COD values.

Temperature in k	$10^4 k$ Sec ⁻¹	COD Values in mg/l	
		Before degradation	After degradation
288	0.30	624	64
298	0.42	624	48
313	0.53	624	48
323	0.61	624	16



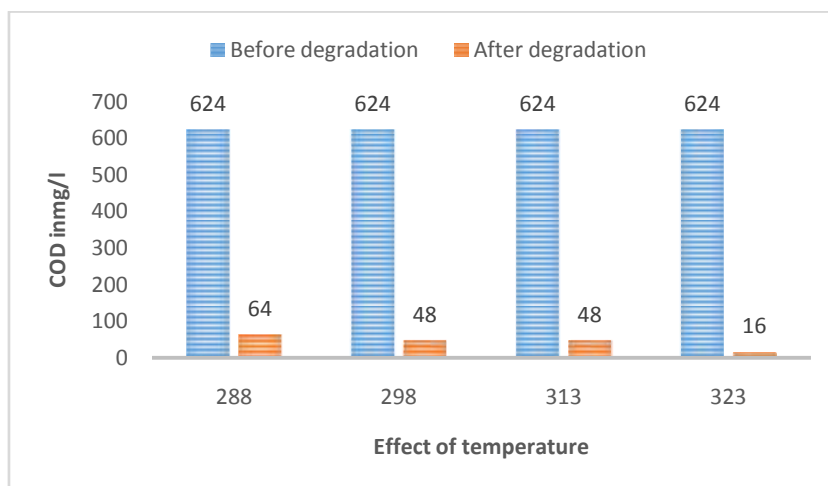


Figure 6 and 7: Effect of temperature of IC on the rate of degradation and COD Values

Table 4: Thermodynamic parameters for the degradation of IC

Temperature in K	Ea	ΔH	ΔS	ΔG
288	4.105 Cal	14.77 KJ/mol	-265.36 J/K	91.20 KJ/mol
298		20.80 KJ/mol	-245.06 J/K	93.83 KJ/mol
313	4.193 Cal	17.28 KJ/mol	-258.86 J/K	98.30 KJ/mol
323		17.27 KJ/mol	-260.66 J/K	101.46 KJ/mol

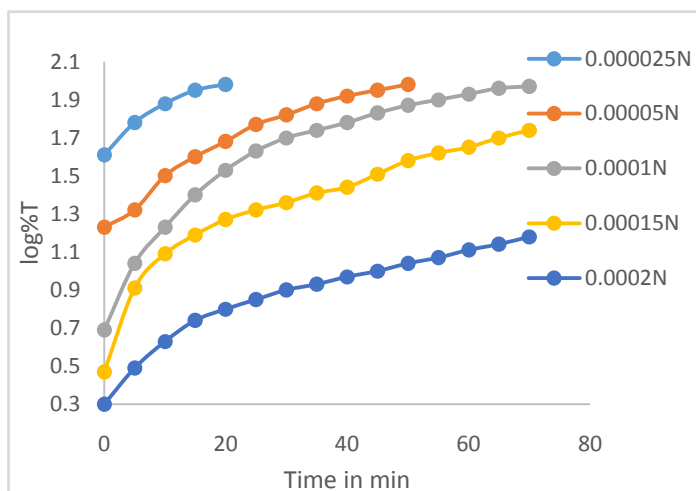
3.2 Reaction with Ruthenium graphite Modified (RuGME) as anode electrode

3.2.1 Effect of Indigo carmine

The reaction has been carried out in the presence of IC (0.0001M), by keeping current constant (4.1mA). The change in concentration of the Indigo carmine was recorded by change in color using spectrophotometer. A plot of $\log T$ (percentage transmittance of light) versus time was linear upto 60% Reaction indicating disappearance of IC follows first order kinetics. The reaction rate decreased with increase in [IC] and the values of rate constants were higher compared to graphite electrode as anode alone. The pH value before and after the electrolysis indicates slight increase towards alkaline pH. The COD for Indigocarmin solution before and after electrolysis were measured. (Table-5, Fig-7 and 8).

Table 5: Effect of [IC] on the rate of degradation and COD values for RuGME electrode

$10^4 [IC]$	$10^4 k$ Sec ⁻¹	Effect of pH		COD Values in mg/l	
		Before degradation	After degradation	Before degradation	After degradation
0.25	6.98	6.23	6.28	464	16
0.5	5.75	6.54	6.58	512	16
1.0	5.87	6.96	7.01	624	32
1.5	5.37	7.13	7.17	816	16
2.0	4.06	7.32	7.36	992	48



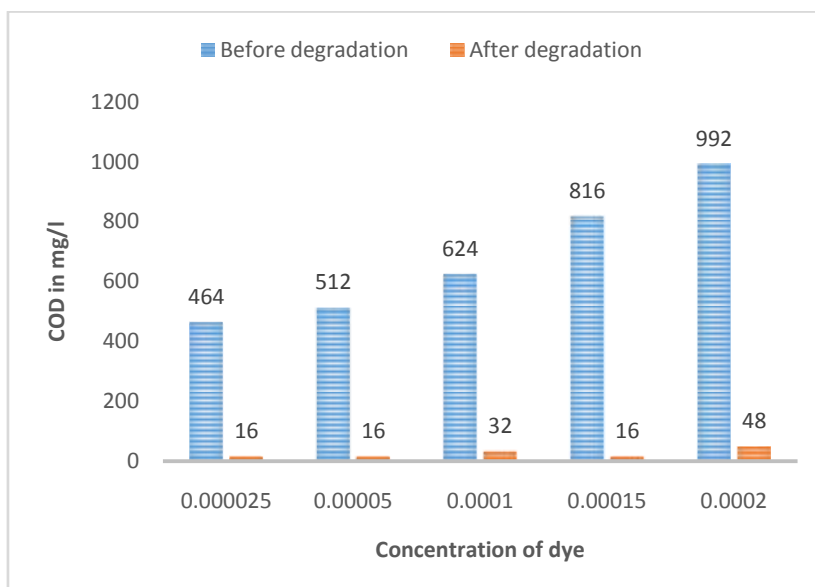


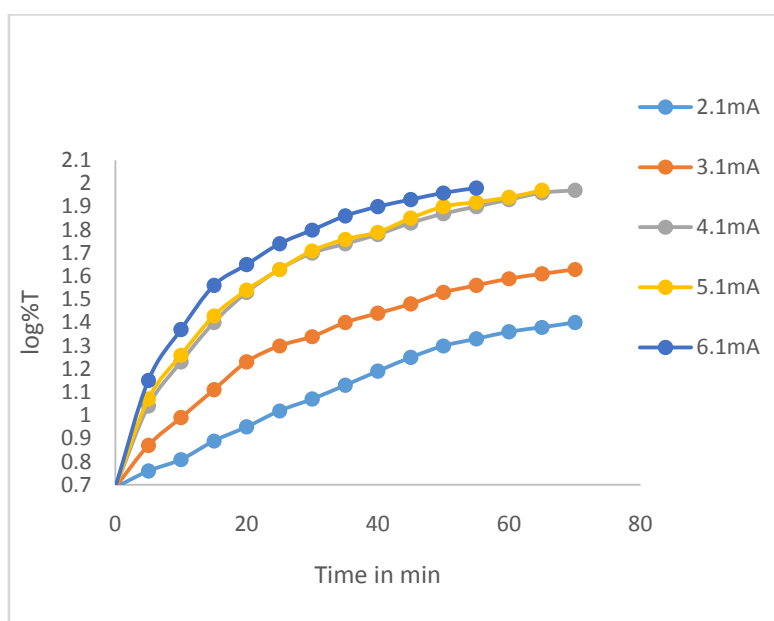
Fig. 8 and 9: Effect of concentration of IC dye on the rate of degradation and cod values

3.2.2 Effect of current on the rate

At fixed [IC] the rate of reaction increased with increased in applied current. The current is varied from 2.1 mA-6.1 mA. The rate of degradation is higher compared to graphite electrode as anode. The COD for different current of Indigocarmine solution before and after electrolysis were measured. (Table-6, Fig-9 and 10)

Table 6: Effect of current on the rate of degradation and COD values.

Current in mA	$10^4 k$ Sec ⁻¹	COD Values in mg/l	
		Before degradation	After degradation
2.1	4.06	624	16
3.1	4.72	624	16
4.1	5.87	624	32
5.1	6.63	624	48
6.1	7.44	624	32



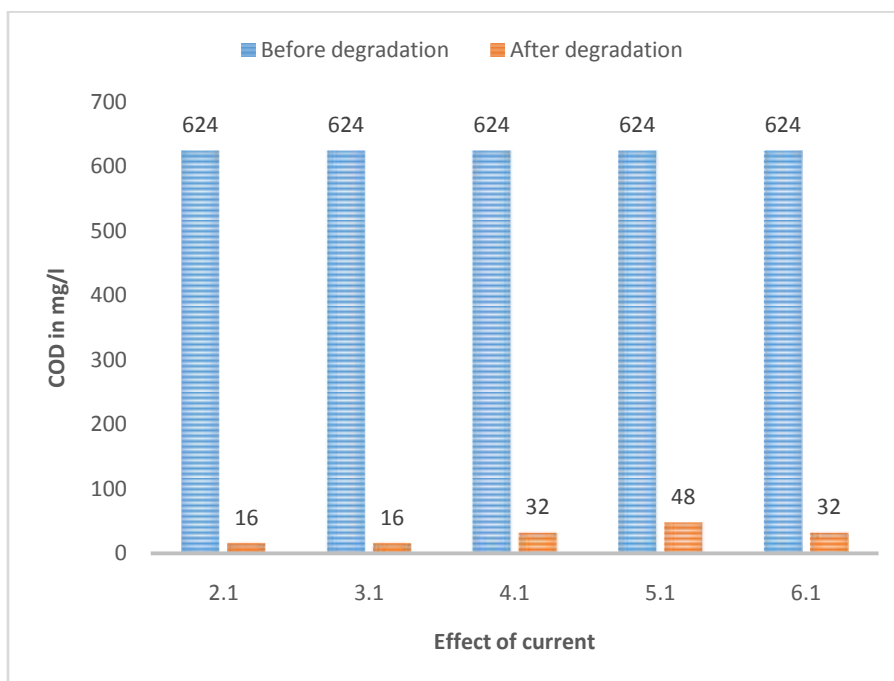


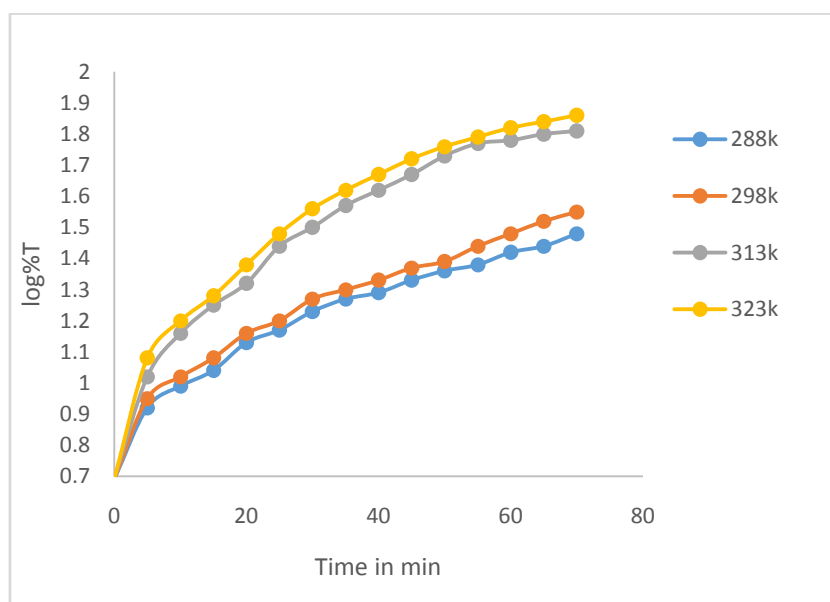
Figure 10 and 11: Effect of current of IC on the rate of degradation and COD Values

3.2.3 Effect of temperature

It is clear that as the temperature increases, the dye degradation is observed. The rate of removal of color is not very significant at low temperature. However, the reaction is more significantly influenced at higher temperature (Table 7). Since the diffusion rate is increased with temperature, an increase of temperature could bring about an increase in the degradation rate.

Table 7: Effect of Temperature on the rate of degradation and COD values

Temperature in k	$10^4 k$ Sec ⁻¹	COD Values in mg/l	
		Before degradation	After degradation
288	3.64	624	16
298	3.91	624	32
313	5.33	624	16
323	5.41	624	16



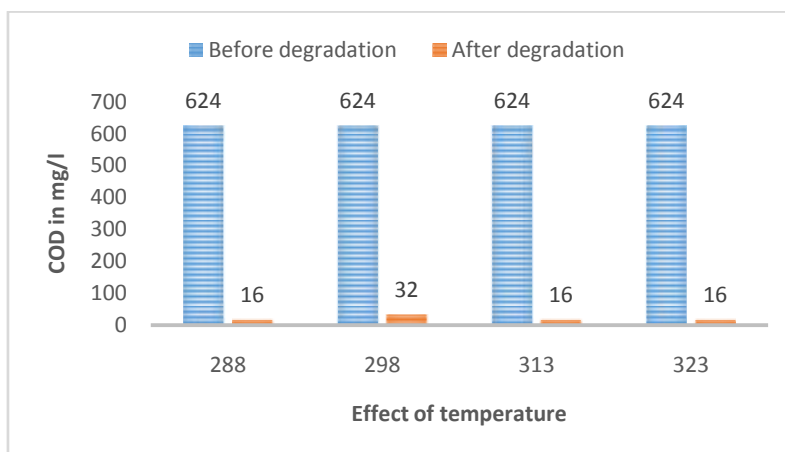


Figure 12 and 13: Effect of current of IC on the rate of degradation and COD Values

Table 8 : Thermodynamic parameters for the degradation of IC for RuGME electrode

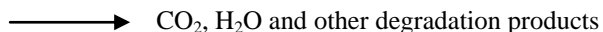
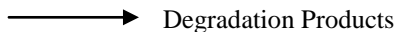
Temperature in K	Ea	ΔH	ΔS	ΔG
288	4.214 Cal	15.34 KJ/mol	-257.23 J/K	89.42 KJ/mol
298		15.15 KJ/mol	-259.32 J/K	92.42 KJ/mol
313	4.241 Cal	15.14 KJ/mol	-259.61 J/K	96.40 KJ/mol
323		15.05 KJ/mol	-261.54 J/K	99.53 KJ/mol

3.2.4 Production of OH radical

The degradation of Indigocarmine solutions were carried out by anodic oxidation. Further, the experiment is carried out in the presence of electrodes with Pt as cathode and graphite as anode. In the electrochemical technique .OH formed by direct electrolysis absorbed as intermediate at the surface of high O₂- overvoltage anode from oxidation of water.



The degradation of IC takes place due to sequential electrochemical reduction and oxidation. Graphite electrodes have high affinity for IC with implication that it will adversely affect electrode performance by competitive adsorption and interaction such as electron shuttling may increase the reactivity. The strong oxidizing agent .OH Radical, the sequential reduction/oxidation with graphite and Pt electrodes which reacts with IC converting into CO₂, H₂O and inorganic ions until their total mineralization is reached. It has been found that Pt electrode possesses greater O₂- over voltage (+0.77V), thus generating higher amount of oxidant OH.. A clear solution is obtained after degradation indicates the absence of formation of insoluble metal oxides. This suggests that oxidizing intermediates which are formed resulting in destruction of the dye. Considering the graphite electrode (GE) advantages such as commercial availability, low costs and easy of modification [22]. The present method can be used for remediation of waste water.



Indigocarmine

Isatin

Fig. 14: Degradation pathway of Indigo carmine

3.2.5 Effect on COD of duration of treatment

The effect of rate of dye degradation on COD was determined. During the experiment the initial COD 624mg/l which gave rise to 48mg/l of COD, a 95% reduction in COD was achieved. The COD level obtained after degradation was within the permitted limit of 250mg/l. A graph showing COD reduction versus treatment time is shown in figure (13).

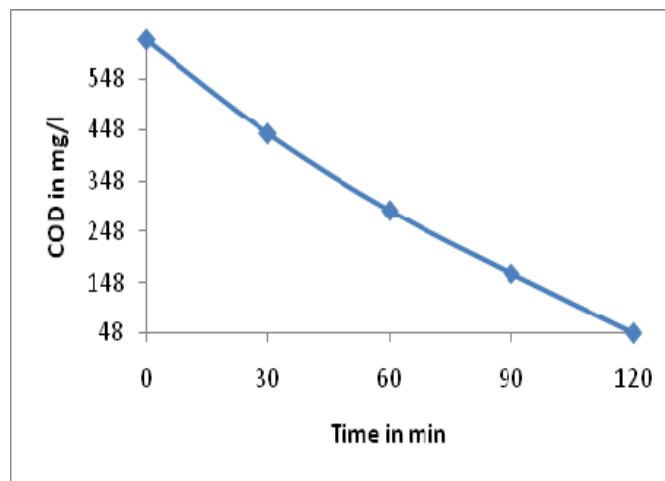


Fig.15: The rate of decrease of COD at different intervals of time.

The effect of current density, [dye] and COD was measured. In all these cases the COD level decreased more than 90 %.The instantaneous current efficiency (ICE) for the anodic oxidation of Indigocarmine dye was calculated from the values of the COD using the equation

$$ICE \quad (\%) \quad = \frac{(COD_i - COD_f)}{8It} \times FV \quad [7]$$

Here; COD_i and COD_f are the chemical oxygen demands before and after degradation respectively, 'I' the current, 'F' the Faraday constant, 'V' the volume of the electrolyte, 't' the treatment time and '8' is the oxygen equivalent mass (geq-1). The ICE data in table indicates, the efficiency of the process linearly related to ICE and indicates higher efficiency of the process for RuMGE anode compared to GE anode

Table 9: ICE values at different experimental conditions for Graphite Electrode

	Variables	ICE
Concentration of Dye 10^{-4}	0.25	188.29
	0.5	182.40
	1.0	165.87
	1.5	161.39
	2.0	156.91
Current in mA	2.1	228.17
	3.1	181.98
	4.1	165.87
	5.1	147.38
	6.1	150.28
Temperature in K	288	102.97
	298	121.04
	313	135.57
	323	162.61

Table10: ICE values at different experimental conditions for RuGME electrode

	Variables	ICE
Concentration of Dye 10^{-4}	0.25	1318.0
	0.5	583.7
	1.0	497.63
	1.5	523.03
	2.0	462.88
Current in mA	2.1	582.06
	3.1	473.16

	4.1	497.63
	5.1	457.63
	6.1	468.26
Temperature in K	288	298.13
	298	387.04
	313	477.06
	323	717.50

3.2.6 Kinetics of Degradation

The degradation of dye taken place in the absence of electrolyte HCL or NaOH. The degradation of dye depends with [dye], current and the concentration of Surface active sites[S]. Since [S] remains constant, the rate of degradation in the present case is given by

$$-\frac{dc}{dt} = \frac{k[I]}{[dye]}$$

The rate constant for the disappearance of Indigocarmine for the degradation process was determined by plotting logT versus time (t), indicating kinetics follows 1st order reaction. Here ‘T’, is percentage transmission of the light obtained from spectrophotometer, at λ_{max} 610nm and it is inversely proportional to the concentration of the dye. The 1st straight line was obtained for 60% of the degradation reaction and afterwards deviation in linearity was observed.

3.2.6 Reuse of Ruthenium graphite Modified (RuGME)

The possibility of reusing the Ru/graphite modified electrode was tested to see the cost effectiveness of the method adopted. After the degradation of the dye, the graphite modified electrode was thoroughly washed with double distilled water and then reused for the degradation by taking fresh dye solution. From the degradation study it was observed that the reuse of the graphite modified electrode to degrade the dye solution showed lesser efficiency.

3.2.7 UV-Visible spectra

Figure 16 (A and B) represents UV-Visible spectra during the electrochemical degradation of Indigocarmine dye. A broad visible colour absorption spectra at 610nm was completely removed at the end of the degradation process. However, the absorption in the UV-region indicates less intense small peak which shows the presence of a minute concentration of organics (<48mg/l COD).

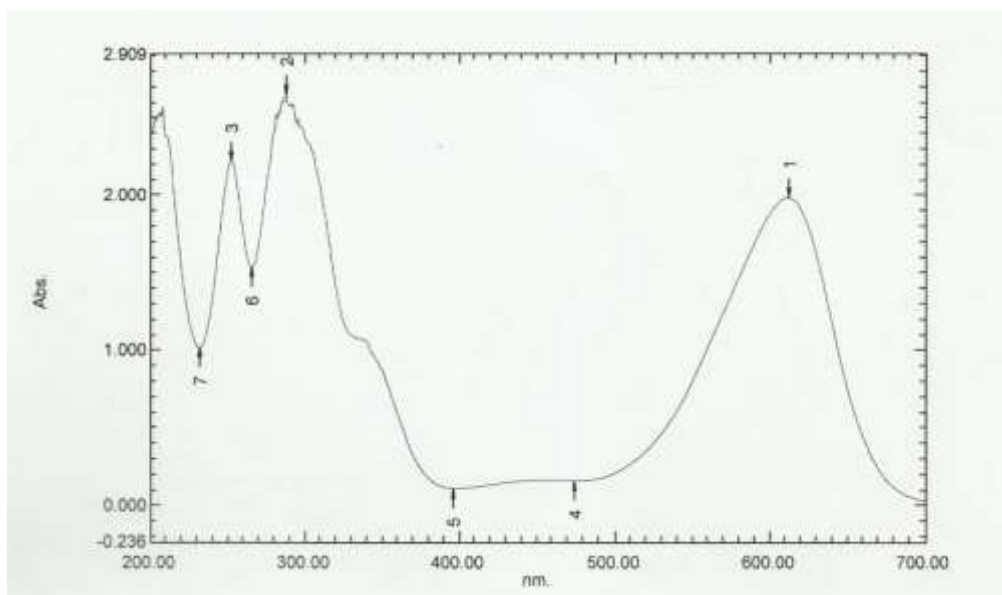


Fig. 16 (A): UV-Visible spectra of IC before degradation

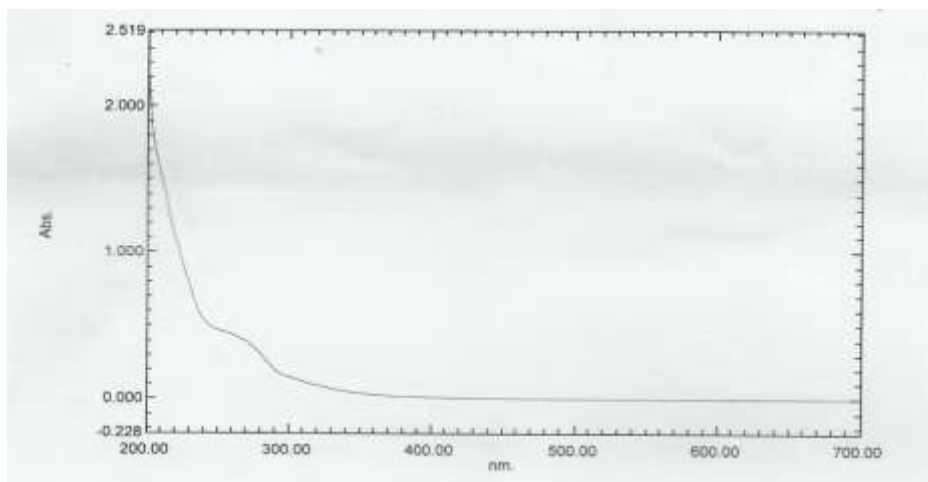


Fig. 16 (B): UV-Visible spectra of IC after degradation.

3.2.8 IR Spectra

FTIR of the compound before degradation is shown in **Figure 12**. There are 12 prominent signals indicating the functional groups. At 3307 cm^{-1} a frequency of NH stretch is shown and at 2968 cm^{-1} , a prominent frequency of OH stretching is seen. Frequency indicating the presence of keto group was at 1662 cm^{-1} . Three prominent peaks of Carbon linkages are shown in the at 1638, 1613 and 1472 cm^{-1} corresponding to isolated Carbon linking with carbon in double bond. In an aromatic ring in c-c double and single bond respectively. Sulphur related peaks were found at 1320 and 1195 cm^{-1} corresponding to Sulfur oxygen double bond linkage and carbon- sulfur linkage. Frequency at 1195 and 893 cm^{-1} corresponds to carbon nitrogen bond and nitrogen to hydrogen bond (-NH secondary amine). At 822 and 732 cm^{-1} indicate the presence of Aromatic CH stretching bond and Aromatic CH bond which is out of plain bending respectively.

FTIR of the compound after degradation is shown in Figure 13. After the degradation of compound three prominent peaks of OH bonds are seen. At 3354 and 2015 cm^{-1} .OH stretch is seen where one is asymmetric stretch and the other is symmetric stretch respectively. The third peak at 1642 indicates the bending of OH group.

Before degradation of IR spectra

FT-IR (KBr) = ν = 3307(-NH), 2968(-OH), 1662(-C=O), 1638(Iso-C=C), 1613 (Ar-C=C), 1472(Ar-C-C), 1320(-S=O), 1195(-C-N), 1032(-C-S), 893(N-H-Wag), 822(Ar-CH (M)), 732(Ar-CH (O)) cm^{-1}

After degradation of IR spectra

FT-IR (KBr) ; ν = 3354(-OH Asy stretch), 2154 (-OH Sym stretch), 1642(-OH, bent) cm^{-1}

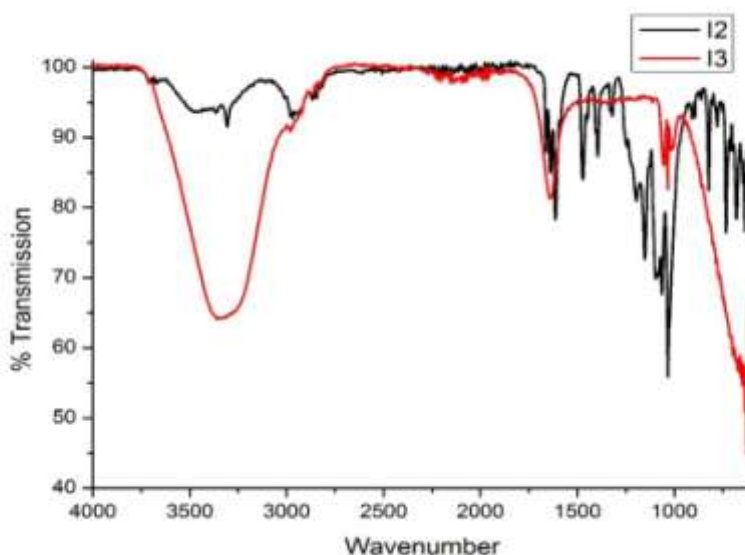


Fig. 17: FT-IR spectra of before degradation and after degradation

3.2.9 Scanning Electron Microscopy (SEM)

Fig 18(a-b) shows the SEM image of Graphite flake and its corresponding EDX profile has shown. From the SEM micrograph, the layered and homogenous nature of graphite was observed in different diameter. The observed size of graphite flake was ranging from 10 to 50 μm . From the EDX profile, the presence of only carbon content indicated the graphite present in the pure form.

Fig 19(a-b) shows the SEM and EDX profile of ruthenium encapsulated in graphite rod. The SEM image of ruthenium encapsulated graphite rod can clearly distinguish from graphite rod. Further, the heterogeneous block dots were observed on the surface, which depict the ruthenium doped or encapsulated inside the graphite rod. The corresponding EDX plot as shows in fig 16(b). From the EDX plot, we clearly noticed the presence of ruthenium peaks in different oxidation states along with graphite carbon peak.

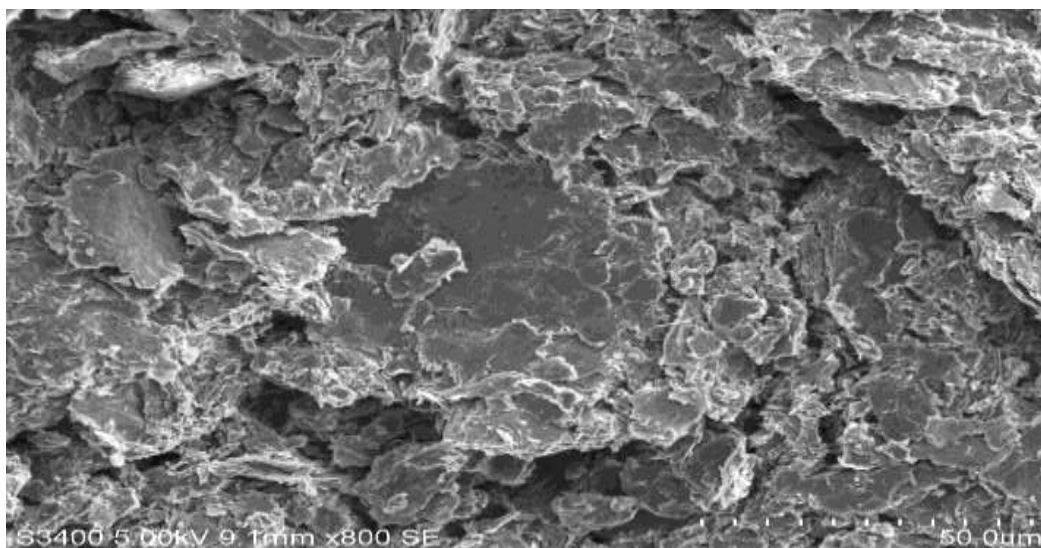


Fig. 18(A): SEM micrographs of graphite Electrode

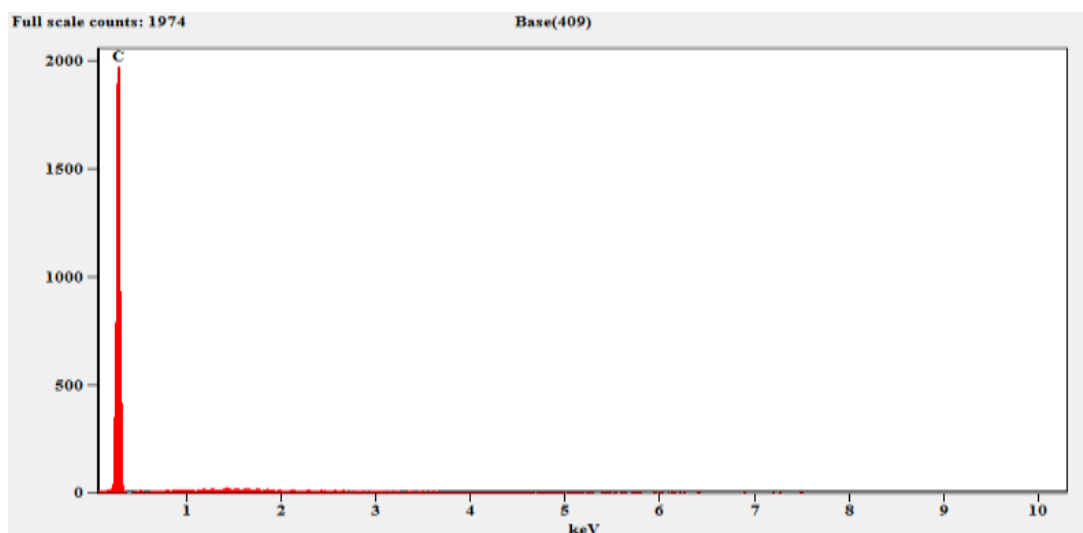


Fig. 18(B): Energy –dispersive X-ray spectroscopy (EDS) for graphite Electrode

Table11: Quantitative results for graphite Electrode

Element Line	Weight %	Weight % Error	Atom %
C K	100.00	± 1.09	100.00
Total	100.00		100.00

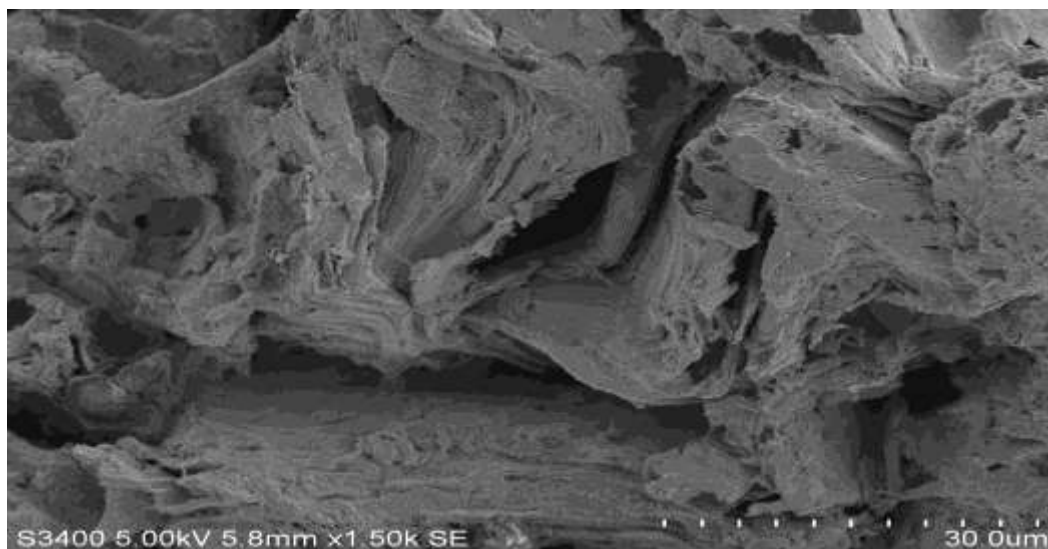


Fig. 19(A): SEM micrographs of Ruthenium graphite modified electrode

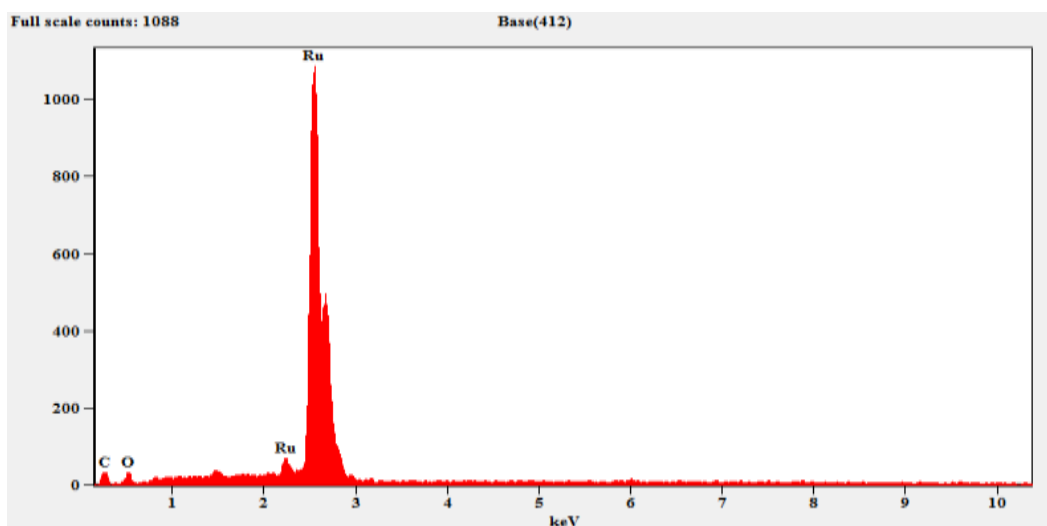


Fig. 19(B): Energy –dispersive X-ray spectroscopy (EDS) for Ruthenium graphite modified electrode

Table12: Quantitative results for Ruthenium graphite modified electrode

Element Line	Weight %	Weight % Error	Atom %
C K	---	---	---
O K	10.29	± 0.72	42.00
Ru L	89.71	± 1.51	58.00
Ru M	---	---	---
Total	100.00		100.00

IV. Conclusion

Electrochemical oxidation in presence of graphite and Ruthenium modified graphite electrode is capable of destroying the chromophore groups of dye found in industrial effluents at short treatment times with low energy consumption and can be reuse the graphite electrode. This method can be applied to the remediation of wastewater containing dyes and organics .Hence the method is cost effective. The rates of Indigo carmine elimination and COD removal were higher on the Ru/ graphite modified electrode compare to that of the graphite electrode.

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