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Properties of Acetophenone
Ethylchloroacetate Binary Mixture

Highlights

Controller Parameters Optimization
Levenberg - Marquardt's Algorithm

Discovering Thoughts, Inventing Future

VOLUME 15 ISSUE 1 VERSION 1.0

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Prediction of Volumetric and Viscometric Properties of Acetophenone – Ethylchloroacetate Binary Mixture at 303K & 323K with different Model Analysis

By M. Sathiyamoorthy & Dr. Mazda Biglari
Higher Colleges of Technology, India

Abstract- In this present investigation the volumetric properties and viscosity of the acetophenone ethylchloroacetate liquid binary mixture were determined. The properties were found as a function of mole fraction and at a temperature of 303 K and 323 K. The excess molar volumes and ultrasonic velocity are also determined. It is used to predict the intermolecular interactions in the process calculation, pipe design and automobile fuel section. The kinematic viscosities of this mixture were analyzed with four different models namely McAllister, Krishnan-Laddha, Jouyban-Acree and Redlichkister. The different properties were plotted against mole fraction of the liquid mixtures at various compositions and temperatures.

Keywords: *viscosity, density, ultrasonic velocity, acetophenone, ethylchloroacetate, volumetic properties.*

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Keywords: viscosity, density, ultrasonic velocity, acetophenone, ethylchloroacetate, volumetic properties.

I. Introduction

Liquid mixtures have attracted considerable attention due to their unusual behavior. In chemical process industries, material are normally handled in fluid form and as the consequence, the physical, chemical, and transport properties of fluids assume importance. Fluid mixtures in process industries are often separated into their components by mass transfer operations. Design of such operations requires quantitative estimation of the properties of fluid mixtures. Recently there has been considerable progress in the studies on intermolecular interactions and the internal structure of liquid mixtures. Thus data on some of the properties associated with the liquids and liquid mixture like density, viscosity and ultrasonic velocity find extensive application in solution theory and molecular dynamics. These results are necessary in chemical, electrochemical, biochemical and kinetic studies.

a) Thermo Physical Properties

Thermo physical properties of liquid mixtures have extensive practical applications in day to day life. Any problem connected with heat, momentum and mass transfer entails knowledge of thermo physical properties and their variation with temperature.

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The data on some of the thermo physical properties associated with the liquids and liquid mixtures find applications in solution theory and molecular dynamics (Mchaweh et al 2004). These results are necessary for interpretation of data obtained from thermo chemical, electrochemical, biochemical and kinetic studies (Kenart et al 2000). These are needed in many engineering problems such as process calculations, simulations and pipe design and automobile fuel selection. The automobile fuel has to be checked for the consistency of its properties before it is supplied to the engine. The thermo physical properties of liquid mixtures like density, viscosity, refractive index, surface tension and ultrasonic velocities are often applied for calculations of other parameters characterizing binary and ternary liquid mixtures.

i. *Density*

Density belongs to the group of most useful intensive physiochemical properties widely applied studies of pure liquids and liquid mixtures. It behaves as an additive volumetric property for ideal solutions. Results of many experimental works show that the analysis of deviation from density as a function of the composition of the mixture is more useful for studies of intermolecular interactions in liquid mixtures that the analogous examination of changes of density. The knowledge of density of liquid mixtures is necessary for calculations of other properties like viscosity and thermo acoustical parameters.

ii. *Viscosity*

Viscosity is not a simple additive property. It is an important transport property for process design in petroleum, petrochemical and other chemical industries involving fluid transportation, mixing, agitation, filtration, heat exchange and concentration. The investigation of viscosity can be a powerful tool for characterization of interactions present in the mixtures.

iii. *Ultrasonic Velocity*

The ultrasonic studies are extensively used to estimate the thermodynamic properties and predict the intermolecular interaction of liquid mixtures. Ultrasonic velocity of binary liquid mixtures could be related either to size and shape of the molecules or to the entropy

effect because of volume and space filling effects with mixing processes. The principle used in the measurement of velocity is based on the accurate determination of the wavelength in the medium. The high frequency generator generates variable frequency, which excites the quartz crystals. The excited quartz crystal generates ultrasonic waves in the experimental liquid. The liquid will serve as an acoustical grating element when ultrasonic waves passes through the ruling of the grating, successive maxima and minima occurs, satisfying the condition for diffraction. Ultrasonic waves are high frequency mechanical waves. Ultrasonic velocity in a medium depends inversely on density and the compressibility of the medium. The variation in the ultrasonic velocity of the liquid mixtures increases or decreases of intermolecular free length of mixing and vice versa.

iv. *Intermolecular Forces*

Intermolecular forces are electrostatic forces of attraction that exist between an area of negative charge on one molecule and an area of positive charge on a second molecule. Intermolecular forces are a secondary method of holding a solid state structure together. As the name implies, these are forces that exist between molecules. Bonds exist within molecules. For reasons that will not be discussed here, intermolecular forces are only associated with systems that use covalent bonding within the molecules. Intermolecular forces are not encountered in systems that employ ionic bonding. Some elements, such as the noble gases, exist with intermolecular forces and no bonding at all. Intermolecular forces exist in three different levels of strength. The differing strengths are a function of the magnitude of the areas of charge that hold them together. These three different forces are Hydrogen bonding (the strongest), Dipole-dipole forces, London dispersion forces (the weakest). Two of the intermolecular forces are associated with polar structures Hydrogen bonding and Dipole-dipole forces. One of the intermolecular forces is associated with non polar structures is London dispersion forces.

b) Models based on viscosities of pure component

i. *McAllister model*

The Eyring's theory of absolute reaction rates was used to develop a model to predict the viscosity of liquid mixtures. Mcallister (1960) proposed a model which assumes the free energy of activation for viscosity are additive on mole fraction basis.

From Eyring's Theory $\upsilon = h\lambda_1/\lambda_2\lambda_3\lambda^2 e^{\delta G/RT}$

$$Y = (hn/m)\, e^{\delta G/RT}$$

McAllister used the above equation for binary liquid mixtures considering various interactions between the molecules in one plane. The size ratio of the 2 molecules should be less than 1.5.

$$\ln \upsilon = x_1^3 \ln\upsilon_1 + 3x_1^2 x_2 \ln\upsilon_{12} + 3x_1x_2^2 \ln\upsilon_{21} + x_2^3 \ln\upsilon_2 -$$
$$\ln(x_1+x_2m_2/m_1) + 3x_1^2x_2 \ln((2+m_2/m_1)/3) + 3x_1x_2^2 \ln(1+2m_2/m_1)/3) + x_2^3 \ln(m_2/m_1)$$

The above equation is McAllister equation based on three body model. It contains 2 constants namely υ_{12} and υ_{21}. The constants can be evaluated using least square method.

ii. *Kirshnan-Laddha model*

Krishnan and Laddha (1963) have proposed an equation to predict the viscosities of binary liquid mixtures based on Eyring's theory of absolute reaction rates.

$$\ln\upsilon_{mix} = x_1 \ln \upsilon_1 + x_2 \ln \upsilon_2 - 2.303x_1x_2 (A + B(x_1 - x_2)) - \ln(x_{1M1}+x_2M_2) + x_1 \ln M_1 + x_2 \ln M_2$$

The constants can be evaluated if the viscosity data is available for binary system at a particular temperature using least squares method.

iii. *Jouyban-Acree model*

Jouyban proposed a model for correlating the viscosity of liquid mixture at various temperatures.

$$\ln \upsilon_m = x_1 \ln \upsilon_1 + x_2 \ln \upsilon_2 + (x_1x_2/T) \Sigma a_i (x_1 - x_2)^{i`}$$

Where υ_m, υ_1,and υ_2 are the viscosities of the mixture and solvents 1 and 2 at temperature T, respectively. A_i are the model constants.

c) Models based on excess properties

i. *Redlich-Kister model*

Redlich and Kister have proposed an equation to predict deviation in excess values of binary liquid mixtures. The equation is as follows

$$Y = x_1 x_2 \Sigma a_{i-1} (x_1 - x_2)^{i-1}$$

Where, Y refers to V^E or $\delta\eta$

d) Models based on ultrasonic velocity

Sound speed by Jacobson's free length theory "Jacobson (1952) is calculated using the following formula.

$$U^{flt} = k / l_{f(mix)}\rho^{1/2}_{exp}$$

Where k is the Jacobson's constant ($k = (93.875+0.375t)\ 10^{-8}$) and depends only on temperature and $l_{f(mix)}$ is intermolecular free length of mixture.

Vandeal vangeal (1969) ideal mixing relation is compared from the following formula.

$$U^{van} = [(x_1/m_1u_1^2 + x_2/m_2u_2^2)\ (x_1m_1 + x_2m_2)]^{-1/2}$$

Where x_1 and x_2 are mole fractions and u_1 and u_2 is speed of sound of acetophenone and benzene respectively. The sound speed in the mixture is given by impedance dependence relation "shipra and parsania (1995) as

$$U^{idr} = [(\chi_1 z_1 + \chi_2 z_2) / (\chi_1 \rho_1 + \chi_2 \rho_2)]$$

Where χ_i, z_i and ρ_i are the mole fractions, impedance and density of the i^{th} component respectively.

II. Experimental Setup & Procedure

The aim of this research is to measure the density and Viscosity of the Acetophenone Ethylchloroacetate binary mixtures at two different temperatures (303, and 323) K and over the entire composition. These values have been used to calculate the excess molar volume (V^E), deviation in viscosity ($\Delta\eta$). The Viscosity values were fitted to the models of McAllister, Krishnan-Laddha and Jouyban-Acree. The excess values were (like excess molar volumes, deviation in viscosity) fitted to Redlich-Kister type equation to obtain their coefficients and standard deviations. The experimental setup has been shown in the figure 1.

a) Viscometers

Capillary viscometers are the most commonly used instruments for Newtonian liquids. Most glass capillary viscometers are operated by force of gravity. Because of small driving force this class of devices is useful for low viscosity liquids ranging from 0.4 to 16000 centistokes. Glass capillary instruments are low stress instruments the shear stress ranges from 10 to 500 dynes/cm$^{2.}$ The principle of these instruments is derived from viscometer originally used by Ostwald.

The Kinematic viscosities were measured at the desired temperature using Ostwald viscometer as shown in Figure 2. The time was measured with a precision of 0.01s and the uncertainty in the viscosity was estimated to be less than 0.0003mpa.s. Ostwald viscometer was previously calibrated using water. In the viscometer a sample of liquid was charged from tube 1 to bulb C, so that level in the arm stands at the mark. The viscometer with the sample is immersed in a water bath so that it attains the desired temperature. Suction is applied so that liquid is drawn up to mark 'A' through bulb D. The efflux time of the liquid between marks A and B is noted after releasing the vacuum. The liquid mixture was charged into the viscometer. After the mixture had attained bath temperature, flow time has been determined. The above steps were continued and reported. The kinematic viscosity was obtained from the working equation

$$\upsilon = at - b/t$$

Where the two constants a and b were obtained by measuring the flow time t of benzene.

b) Pycnometer

Pycnometer is a vessels with capillary necks in which volume of liquid is weighed. The volume is determined by weighing the vessel filled with water at definite temperature. Densities were determined by using 25cm^3 bicapillary Pycnomete. The quantity of liquid is adjusted so that the liquid meniscus is at the mark on the horizontal capillary while the other arm is completely filled. Tilting the completely filled unit slightly makes this adjustment and drawing liquid slowly from other capillary by touching a piece of filter paper to it. The Pycnometer filled with air bubble free experimental liquids was kept in a transparent walled water bath (maintained constant to ± 0.01K) for 15 minutes to attain thermal equilibrium. The precision density measurements were within ±0.0003g.cm^{-3}.

c) Thermostatic bath

Water bath was used for maintaining a constant temperature during the testing. It was capable of controlling temperature with an accuracy of ±0.01^{0}C.

d) Experimental Procedure

The charge for viscometer was prepared by taking 20 cc of the solution obtained by mixing the two liquids in different proportions. The thermostat was set to the desired temperature. After it had been cleaned and dried the viscometer was immersed in the bath so that the mark A is at least 2 cm below the surface of the bath liquid. The liquid

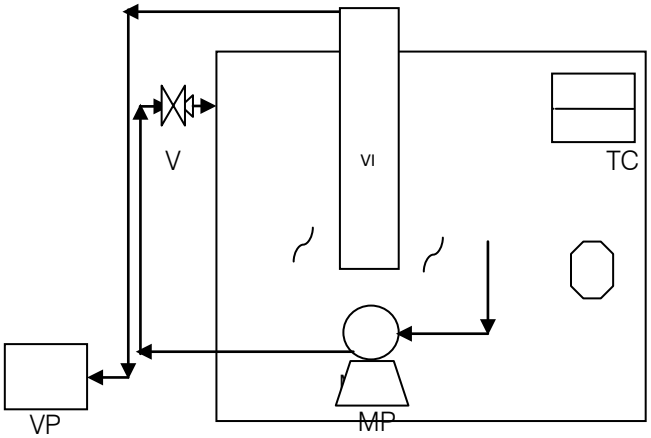


Figure 1 : Experimental setup

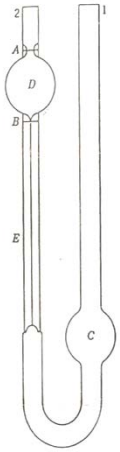


Figure 2 : Oswald viscometer

Figure 3 : Oswald Sprengel pycnometer

VI-Viscometer TC-Temperature controller, V-Control valve MP-Monobloc pump VP - Vacuum pump mixture was charged into tube 1 of the viscometer so that the air bubbles were absent and the level in this arm stood at the mark at the bulb when the temperature was attained. After the sample had attained the bath temperature it was blown up to a point 2cms above the mark A and the liquid was allowed to flow freely and time required for the liquid to flow from top to bottom mark was taken as the flow time. The above steps were continued and an average of 5 sets of flow time was reported. The stop watch used had an accuracy of 0.01 sec.

The chemicals used in this investigation are:

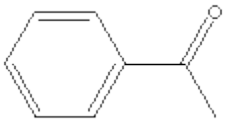
- Acetophenone.
- Ethylchloroacetate.

The purities of the compounds were checked by comparing the measured densities and viscosities with those reported in the literature.

i. *Properties of Chemicals - Acetophenone*

- Acetylbenzene
- 1-Phenylethanone
- Phenyl methyl ketone
- Methyl phenyl ketone

a. Structure



b. Description

Colorless liquid, sweet, almond odor

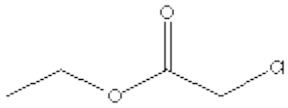
c. Uses

In perfumery to impart an orange-blossom-like odor, catalyst for polymerization of olefins, in organic synthesis, especially as photo sensitizer.

ii. *Properties of Chemicals - Ethylchloroacetate*

- Chloroacetic acid, ethyl ester
- Ethyl Chloro ethanoate

a. Structure



b. Description

Colorless liquid, mobile liquid, extremely irritating, pungent, fruity odor, lachrymator.

c. Uses

Solvent, organic syntheses, military poison, vat dyestuffs.

Table 1 : Properties of pure components

S. No.	Properties	Unit	Acetophenone	Ethylchloroacetate
1	Formula	--	C_8H_8O	$C_4H_7ClO_2$
2	Molar mass	Kg/kmol	120.15	122.55
3	Melting Point	°C	20	-26
4	Boiling Point	°C	201.7	144
5	V.Pressure Saturation	mmHg	0.3	5
6	Concentration	ppm	1300ppm	13160ppm
7	Critical Temp	°C	428	345
8	Critical Press	mmHg	3.8	37.4
9	Density	Kg/m^3	1.027	1.15
10	Solubility in Water	g/L	5.5	Insoluble
11	Viscosity	cp	2.28	2.93×10^{-3}
12	Surface Tension	g/s^2	39.8	31.7
13	Refractive index	--	1.5339	1.4235
14	Heat of Vaporization	kJ/mol	49.0	49.4

III. Results & Discussions

Assuming the shapes of the molecules are spherical, the size ratio of the molecules is given by the formula

$$r_1/r_2 = [(M_1/M_2)(\rho_2/\rho_1)]^{1/3}$$

For McAllister model the size ratio should be less than 1.5.

Where,

r_1, r_2 are the radius of the component 1 and 2

ρ_1, ρ_2 are the density of the pure component 1 and 2

M_1, M_2 are the molecular mass of component 1 and 2

Ethylchloroacetate/Acetophenone =0.9897

Since the size ratio for binary system is less than 1.5, so the system was considered for McAllister three-body model. McAllister model, Krishnan and Laddha model were tested with experimentally obtained viscosity data for the binary and ternary mixtures at 30^0C, 50^0C for the following mixtures.

Ethylchloroacetate (1) + Acetophenone (2)

The respective binary and ternary constants ν_{12}, ν_{21}, were determined by the method of least squares for each system. With these constants viscosity was then calculated for binary and ternary systems at each composition. The deviation of experimental value from the predicted was calculated as follows.

$$\text{Percentage Deviation, } d = ((\nu_{exp}-\nu_{calc})/\nu_{calc})*100$$

Standard Deviation was calculated using the relationship,

$$SD = (\Sigma (\nu_{exp} - \nu_{calc})^2/ (N-m)^{1/2}$$

Where,

N-Number of data points, m – Number of coefficients

The binary viscosity and density values are used to calculate viscosity deviation using the relationship

$$\Delta\eta = \eta 12-(x_1\eta_1 + x_2\eta_2)$$

The density values have been used to calculate excess molar volume using the formula

$$V^E = (x_1m_1 + x_2m_2) / \rho_{12} - x_1m_1/ \rho_1 - x_2 m_2/ \rho_2$$

Results of Present Investigation show that McAllister model, Joubyan-Acree can be used to predict viscosity of binary mixtures. Redlich- Kister equation can be used to predict Excess molar volume of binary liquid mixtures.

a) Discussion

Deviation of physical property of liquid mixtures from the ideal behavior is the measure of the interaction between the molecules which is attributed to either adhesive or cohesive forces.

McAllister equation, Krishnan-Laddha equation and Jouyban-Acree equation were tested with the experimentally obtained data at 30^0C and 50^0C. The constants were obtained by the method of least squares. With these constants the viscosity values were calculated at each composition. The calculated values agreed with the experimental values with a high degree of precision for McAllister model compared to Krishnan-Laddha model. The viscosity of a mixture strongly depends on entropy of the mixture, which is related to liquid structure and enthalpy; and consequently to molecular interactions between the components of the mixtures.

The variation of $\Delta\eta$ and V^E with mole fraction of Ethylchloroacetate for the system Ethylchloroacetate (1) + Acetophenone (2) was studied at 30^0C and 50^0C respectively. The excess molar volume and deviation in viscosity were fitted with Redlich - Kister type equation.

For the mixtures without strong interactions the viscosity deviations are negative. According to this, V^E is the result of contributions from several opposing effects. This may be divided arbitrarily into three types namely, physical, chemical and structural. Physical effects contribute to positive term of V^E. The chemical or specific intermolecular interactions result in a volume decrease and contribute negative values to V^E. The structural contributions are mostly negative and arise from interstitial accommodation and changes in free volume. The actual volume change therefore depends on the relative strength of these effects.

The V^E values were for found to be negative. Negative values of V^E suggest specific interaction between mixing components. Ethylchloroacetate is a weak dipolar molecule, with a dipolar moment of 2.69. Whereas Acetophenone is strong dipolar molecule, with a dipole moment of 3.028. Oxygen group in Acetophenone is attracted towards the Chlorine group in Ethylchloroacetate, which forms dipole-dipole bond. The negative deviations in viscosity over the whole composition range suggest that in these mixtures, the forces between unlike molecules are lesser than the force between like molecules.

Table 2 : Determination of Excess Molar Volume At 303K

S.No	Molefraction of Ethyl Chloroacetate (x_1)	Density ρ (g/cc)	Excess Volume V^E (cc/gmole)	kinematic Viscosity γ (cS)	Absolute Viscosity η (cP)	Viscosity Deviation Δη (cP)
1	0	0.947	0	1.4969	1.41831	0
2	0.1089	1.000	-4.4368	1.1523	1.15287	-0.2596
3	0.2151	1.002	-2.4388	1.1222	1.12489	-0.2068
4	0.3199	1.006	-0.7810	1.058	1.06580	-0.1887
5	0.4223	1.047	-0.3277	1.015	1.06300	-0.1522
6	0.5231	1.064	-3.0582	0.9312	0.99135	-0.1574
7	0.6224	1.072	-1.8051	0.8809	0.94454	-0.1302
8	0.7191	1.058	1.65118	0.8474	0.89722	-0.0882
9	0.8144	1.085	0.82216	0.8273	0.89786	-0.0339
10	0.9086	1.094	1.8223	0.787	0.86145	-0.000
11	1	1.132	0	0.7164	0.81114	0

In this work density (ρ), viscosity (μ) and ultrasonic velocity (u) of pure acetophenone and ethylchloroacetate as well as binary mixture constituted by these two chemicals at temperatures of 303k and 323k respectively. The literature survey showed that no measurements have been previously reported for the mixture studied in this project.

Table 3 : Determination of isentropic compressibility, molar volume, intermolecular free length at 303 K

Molefraction of ethylchloroacetate (x1)	Density ρ g/cc	Ultra-sonic velocity U ms^{-1}	Isentropic compress-ibility (Ks) $x10^{-10}$ $m^2 N^{-1}$	Molar Volume cm^3/mol	Inter-molecular free length L_f x 10^{-11} m	Acoustical Impedance (Z)x10^6 kg/m^2s
0	0.9475	1478	4.8287	126.860	4.559	1400.78
0.10	1.0005	1430	4.8850	120.390	4.586	1431.11
0.21	1.0024	1472	4.6040	120.405	4.452	1475.53
0.31	1.0069	1396	5.0903	120.107	4.681	1406.43
0.42	1.0473	1364	5.1261	115.698	4.698	1429.35
0.52	1.0646	1340	5.2249	114.036	4.743	1427.41
0.62	1.0722	1403	4.7365	113.435	4.515	1504.58
0.71	1.0588	1324	5.3812	115.086	4.813	1402.69
081	1.0853	1222	6.1662	112.478	5.153	1326.67
0.90	1.0946	1390	4.7256	111.725	4.510	1521.93
1	1.1322	1188	6.2494	108.191	5.187	1346.01

Table 4 : Predicted kinematic viscosities of ethylchloroacetate + acetophenone mixture at 303 K

Molefraction of Ethyl Chloroacetat	u_{Exp} ms^{-1}	u^{IDR} ms^{-1}	u^{VAN} ms^{-1}	u^{FLT} ms^{-1}	% Deviation		
					u^{FLT} ms^{-1}	u^{VAN} ms^{-1}	u^{IDR} ms^{-1}
0	1478	1478.4	1478	1478	$-1.538x10^{-14}$	0	0
0.1089	1430	1441.49	1448	1430	0	-1.227	-0.7698
0.2151	1472	1406.95	1444	1472	0	1.924	4.6229
0.3199	1396	1374.19	1464	1396	$1.628x10^{-14}$	-4.624	1.6451
0.4223	1364	1343.37	1510	1364	0	-9.656	1.5949
0.5231	1340	1314.12	1589	1340	$1.696x10^{-14}$	-15.64	2.0296
0.6224	1403	1286.31	1714	1403	$1.62x10^{-14}$	-18.70	9.0865
0.7191	1324	1260.14	1915	1324	0	-30.81	5.1307
0.8144	1222	1235.18	1435	1222	0	-14.86	-1.0349
0.9086	1390	1211.29	1304	1390	0	6.615	14.786
1	1188	1188.8	1188	1188	0	0	$-1.9x10^{-14}$

Table 5 : Predicted absolute viscosities of ethylchloroacetate + acetophenone mixture at 303 K

Molefraction of Ethyl Chloroacetate (x_1)	γ_{expt} (cS)	γ_{pred} (cS) (McAllister Model)	γ_{pred} (cS) (Jouyban- Acree Model)	γ_{pred} (cS) (K-L model)
0	1.4969	1.5268	1.4969	1.4969
0.1089	1.1523	1.9793	1.1862	1.1864
0.2151	1.1222	1.8463	1.0538	1.0540
0.3199	1.0585	1.3875	1.0666	1.0668
0.4223	1.015	0.9373	1.0478	1.0481
0.5231	0.9312	0.6241	0.9555	0.9558
0.6224	0.8809	0.4437	0.8637	0.8640
0.7191	0.8474	0.3607	0.8286	0.8289
0.8144	0.8273	0.3529	0.8294	0.8296
0.9086	0.787	0.4347	0.7931	0.7933
1	0.7164	0.7164	0.7164	0.7164

Table 6 : Predicted excess molar volume, isentropic compressibility (ks) , intermolecular free length(l_f) by (R-K model) for the mixture at 303 K

u_{Exp} ms^{-1}	u^{IDR} ms^{-1}	u^{VAN} ms^{-1}	u^{FLT} ms^{-1}	% Deviation		
				u^{FLT} ms^{-1}	u^{VAN} ms^{-1}	u^{IDR} ms^{-1}
0	1478	1478.4	1478	1478	$-1.538x10^{-14}$	0	0
0.1089	1430	1441.49	1448	1430	0	-1.227	-0.7698
0.2151	1472	1406.95	1444	1472	0	1.924	4.6229
0.3199	1396	1374.19	1464	1396	$1.628x10^{-14}$	-4.624	1.6451
0.4223	1364	1343.37	1510	1364	0	-9.656	1.5949
0.5231	1340	1314.12	1589	1340	$1.696x10^{-14}$	-15.64	2.0296
0.6224	1403	1286.31	1714	1403	$1.62x10^{-14}$	-18.70	9.0865
0.7191	1324	1260.14	1915	1324	0	-30.81	5.1307
0.8144	1222	1235.18	1435	1222	0	-14.86	-1.0349
0.9086	1390	1211.29	1304	1390	0	6.615	14.786
1	1188	1188.8	1188	1188	0	0	$-1.9x10^{-14}$

Table 7 : Predicted ultrasonic velocity deviation for A Ethylchloroacetate+Acetophenone mixture at 303 K

Molefraction of Ethyl Chloroacetate (x_1)	γ_{expt} (cS)	γ_{pred} (cS) (McAllister Model)	γ_{pred} (cS) (Jouyban- Acree Model)	γ_{pred} (cS) (K-L model)
0	1.4969	1.5268	1.4969	1.4969
0.1089	1.1523	1.9793	1.1862	1.1864
0.2151	1.1222	1.8463	1.0538	1.0540
0.3199	1.0585	1.3875	1.0666	1.0668
0.4223	1.015	0.9373	1.0478	1.0481
0.5231	0.9312	0.6241	0.9555	0.9558
0.6224	0.8809	0.4437	0.8637	0.8640
0.7191	0.8474	0.3607	0.8286	0.8289
0.8144	0.8273	0.3529	0.8294	0.8296
0.9086	0.787	0.4347	0.7931	0.7933
1	0.7164	0.7164	0.7164	0.7164

Table 8 : Determination of Gibbs free energy for Ethylchloroacetate + Acetophenone mixture

Molefraction of Ethyl Chloroacetate (x_1)	Gibbs free energy at 303K	Gibbs free energy at 323K
0	9.222	-6.3311
0.1089	-457.120	-688.87
0.2151	-326.553	-546.015
0.3199	-279.174	-453.261
0.4223	-194.740	-384.524
0.5231	-224.702	-374.524
0.6224	-180.233	-266.613
0.7191	-98.368	-105.783
0.8144	18.107	-139.395
0.9086	67.268	0.3919
1	-4.204×10^{-5}	-1.142×10^{-5}

Table 9 : Determination of Excess Molar Volume At 323 K

S.No	Molefraction of Ethyl Chloroacetate (x_1)	Density ρ (g/cc)	Excess Volume V^E (cc/gmole)	kinematic Viscosity γ (cS)	Absolute Viscosity η (cP)	Viscosity Deviation $\Delta\eta$ (cP)
1	0	0.92	0	1.3832	1.2831	0
2	0.1089	0.982	-4.7043	0.9647	0.9481	-0.3356
3	0.2151	1.000	-4.36	0.9379	0.9382	-0.2816
4	0.3199	1.005	-2.5525	0.8949	0.9000	-0.2448
5	0.4223	1.042	-4.366	0.8474	0.8832	-21443
6	0.5231	1.22	-4.2074	0.7836	0.8323	-0.2015
7	0.6224	1.069	-2.7041	0.7568	0.8096	-0.1527
8	0.7191	1.055	1.1099	0.7467	0.7881	-0.0892
9	0.8144	1.083	0.3193	0.6828	0.7399	-0.0806
10	0.9086	1.093	1.5366	0.6694	0.7317	-0.0224
11	1	1.130	0	0.6222	0.7031	0

Table 10 : Predicted kinematic viscosities of ethylchloroacetate + acetophenone mixture at 323 K

Molefraction of Ethyl Chloroacetate (x_1)	γ_{expt} (cS)	γ_{pred} (cS) (McAllister Model)	γ_{pred} (cS) (Jouyban- Acree Model)	γ_{pred} (cS) (K-L model)
0	1.3832	1.4108	1.3832	1.3832
0.1089	0.9647	1.7765	0.9768	1.6401
0.2151	0.9379	1.4981	0.8992	1.5207
0.3199	0.8949	0.9798	0.9276	1.2388
0.4223	0.8474	0.5697	0.8590	1.1215
0.5231	0.7836	0.3307	0.7721	1.0689
0.6224	0.7568	0.2111	0.7454	0.9602
0.7191	0.7467	0.1673	0.7427	0.8230
0.8144	0.6828	0.1776	0.7033	0.7314
0.9086	0.6694	0.2693	0.6572	0.6802
1	0.6222	0.6222	0.6222	0.6222

Table 11 : Predicted absolute viscosities of ethylchloroacetate + acetophenone mixture at 323 K

Molefraction of EthylChloroacetate (x_1)	Molefraction of Acetophenone (x_2)	dynamic viscosity at 30 °C (g/cm.s)(η_{mix})	Grunberg-Nissan deviation parameter (d)
0	1	1.2831	0
0.1089	0.8911	0.9481	-2.4435
0.2151	0.7849	0.9382	-1.0878
0.3199	0.6801	0.9000	-0.7456
0.4223	0.5777	0.8832	-0.4900
0.5231	0.4769	0.8323	-0.4735
0.6224	0.3776	0.8096	-0.3662
0.7191	0.2809	0.7881	-0.2719
0.8144	0.1856	0.7399	-0.4012
0.9086	0.09144	0.7317	-0.1819
1	0	0.7031	0

Table 12 : Predicted excess molar volume for ethylchloroaacetate + acetophenone mixture at 323 K

Molefraction of Ethyl Chloroacetate(x_1)	V^E(pred) (R-Kmodel) (cc/gmole)	$\Delta\eta$ (pred) (R-K model)
0	9.222	-6.3311
0.1089	-457.120	-688.87
0.2151	-326.553	-546.015
0.3199	-279.174	-453.261
0.4223	-194.740	-384.524
0.5231	-224.702	-374.524
0.6224	-180.233	-266.613
0.7191	-98.368	-105.783
0.8144	18.107	-139.395
0.9086	67.268	0.3919
1	-4.204×10^{-5}	-1.142×10^{-5}

Table 13 : Parameters of McAllister constant for ethylchloroaacetate + acetophenone at 303 K & 323 K

Temperature	A	B	SD
303K	-2.25	-1.434	0.4662
323K	-30111	-2.307	0.4807

Table 14 : Parameters of the Krishnan and Laddha constants and standard deviations for the viscosity of ethylchloroaacetate + acetophenone at 303 K & 323 K

Temp T (K)	A_0	A_1	A_2	A_3	A_4	A_5	A_6	SD
303K	2.27	-0.8	-3.2	0.79	0.28	0.95	-	0.0406
323K	4.49	3.255	5.099	-3.51	-0.8	0.23	0.2	0.473

Table 15 : Parameters of the Jouyban Acree constants and standard deviations for the viscosity of ethylchloroaacetate + acetophenone at 303 K & 323 K

Temp T (K)	A_0	A_1	A_2	A_3	A_4	A_5	A_6	SD
303K	-1906	605.5	2111	-556	-200	-66	-	0.0370
323K	4605	4645	-3474	3901	47.4	-207	-19	0.0003

Table 16 : Parameters of the Redlich Kister constants and standard deviations for the viscosity of ethylchloroaacetate + acetophenone at 303 K & 323 K

Temp T (K)	A_0	A_1	A_2	A_3	A_4	A_5	A_6	SD
303K	8.89	-6.563	-9.66	6.84	1.32	-0.24	-0.54	0.596
323K	13.0	-8.493	-14.6	9.09	2.46	-0.56	-0.86	0.042

Table 17 : Parameters of the Redlich Kister constants and standard deviations for the excess volume of ethylchloroaacetate + acetophenone at 303 K & 323 K

Temp T (K)	A_0	A_1	A_2	A_3	A_4	A_5	A_6	SD
303K	207	-187	-298	208.02	103.2	-19.4	-11.9	1.214
323K	173	-179	-236	192	80.17	11.51	16.61	0.993

Table 18 : Parameters of the Redlich Kister constants and standard deviations for the Isentropic compressibility (Ks), intermolecular free length (Lf) of ethylchloroaacetate + acetophenone at 303 K & 323 K

	A_0	A_1	A_2	A_3	A_4	A_5	A_6	SD
K_S^E x 10^{-10} m^2 N^{-1}	-358	-21	393.2	21.67	-57	-0.5	22.44	3.56
L_f^E X10-11m	-353	-18	397.9	20.57	-65	-2.3	21.15	3.13

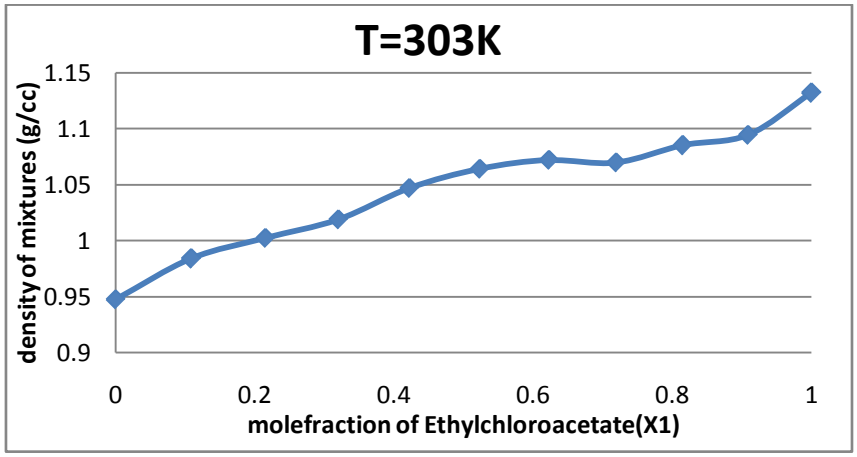


Figure 4 : Plot of mole fraction of ethylchloroacetate Vs density of mixtures at 303K

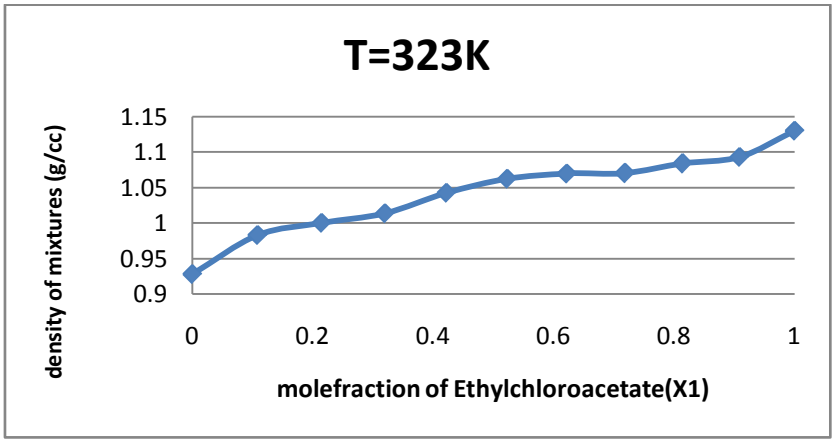


Figure 5 : Plot of mole fraction of ethylchloroacetate Vs density of mixtures at 323K

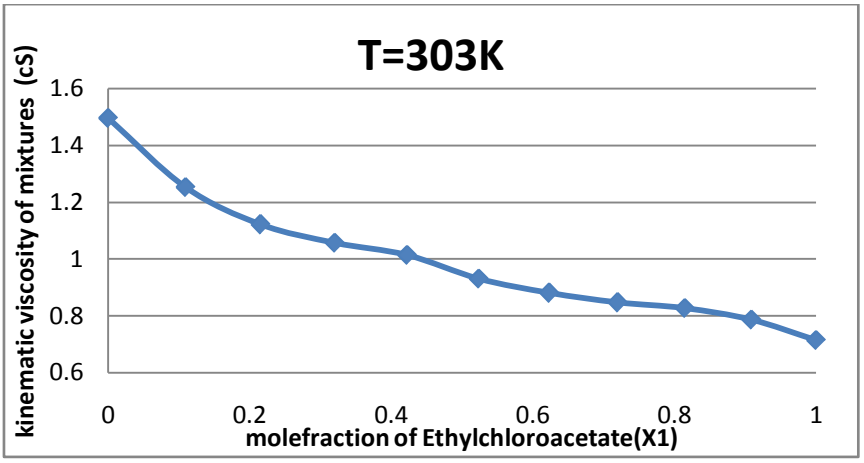


Figure 6 : Plot of mole fraction of ethylchloroacetate Vs viscosity of mixtures at 303K

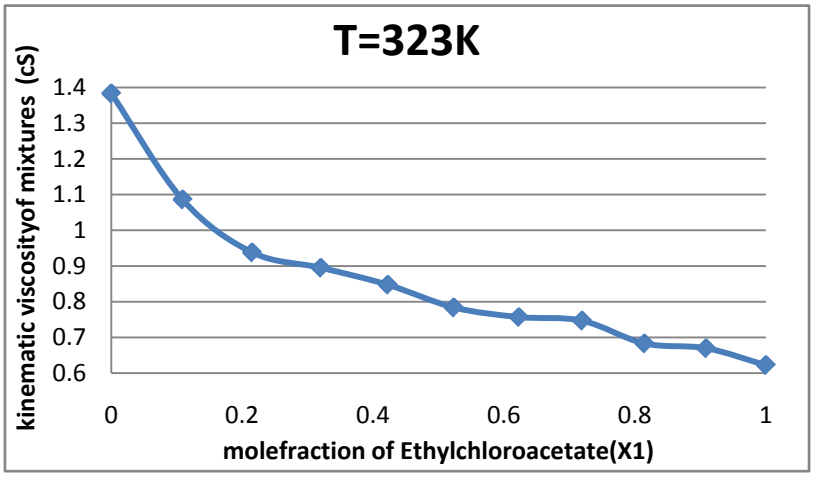


Figure 7 : Plot of mole fraction of ethylchloroacetate Vs viscosity of mixtures at 323K

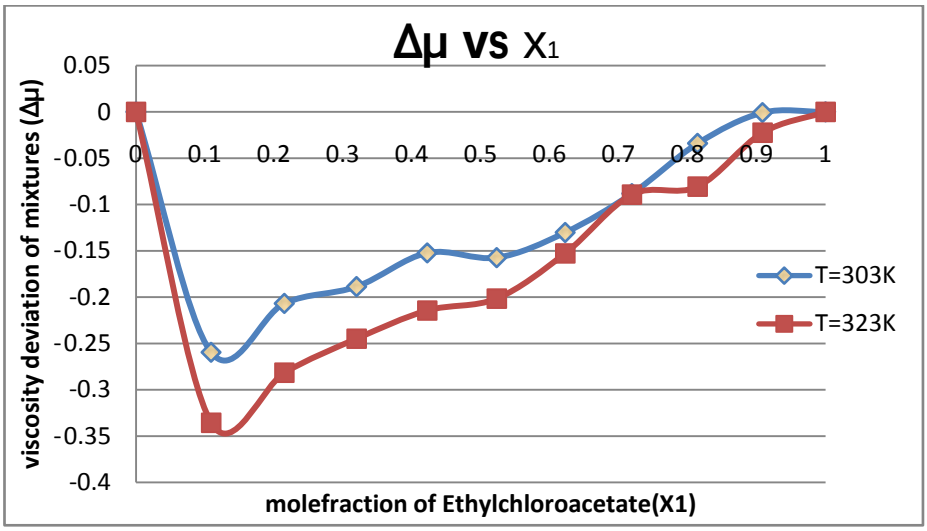


Figure 8 : Plot of mole fraction of ethylchloroacetate Vs viscosity deviation at 303K & 323K

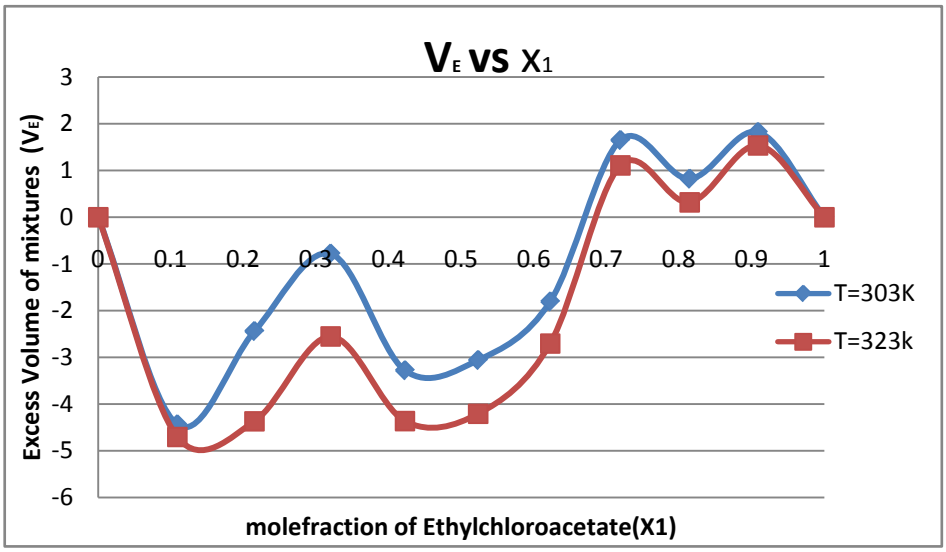


Figure 9 : Plot of mole fraction of ethylchloroacetate Vs excess volume at 303K & 323K

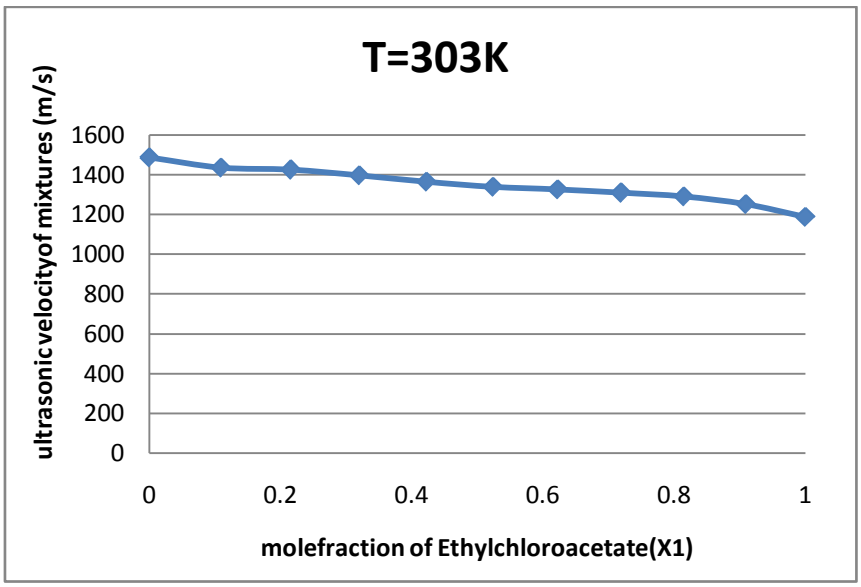


Figure 10 : Plot of mole fraction of ethylchloroacetate Vs ultrasonic velocity 303K

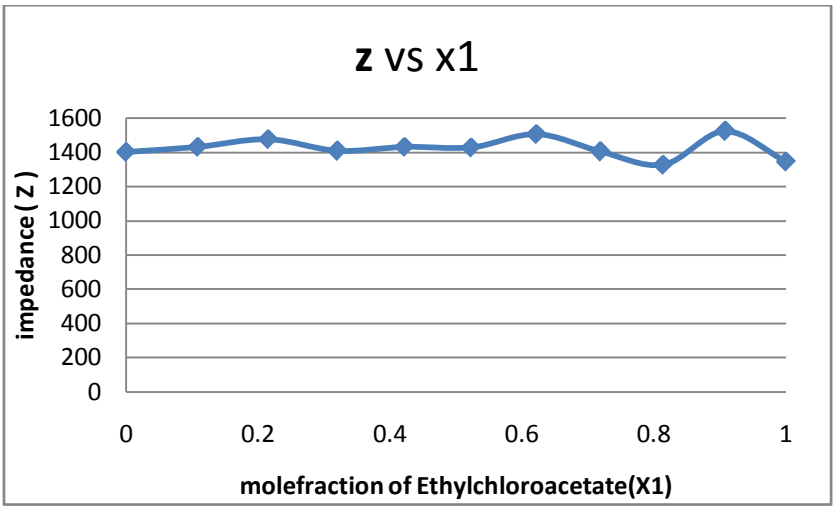


Figure 11 : Plot of mole fraction of ethylchloroacetate Vs Impedance

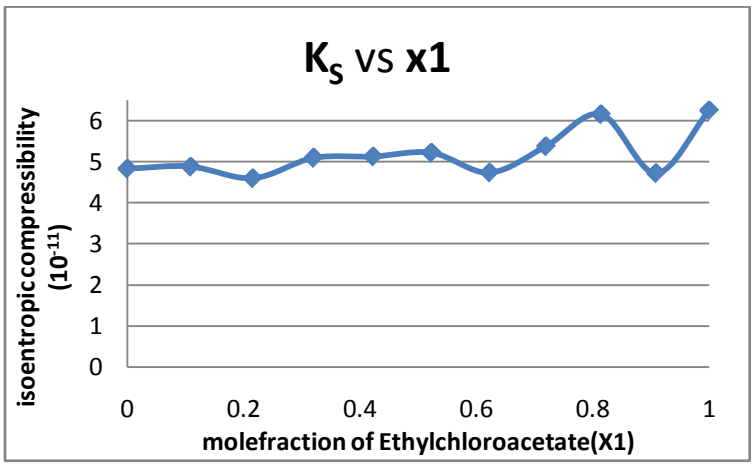


Figure 12 : Plot of mole fraction of ethylchloroacetate Vs Isentropic compressibility

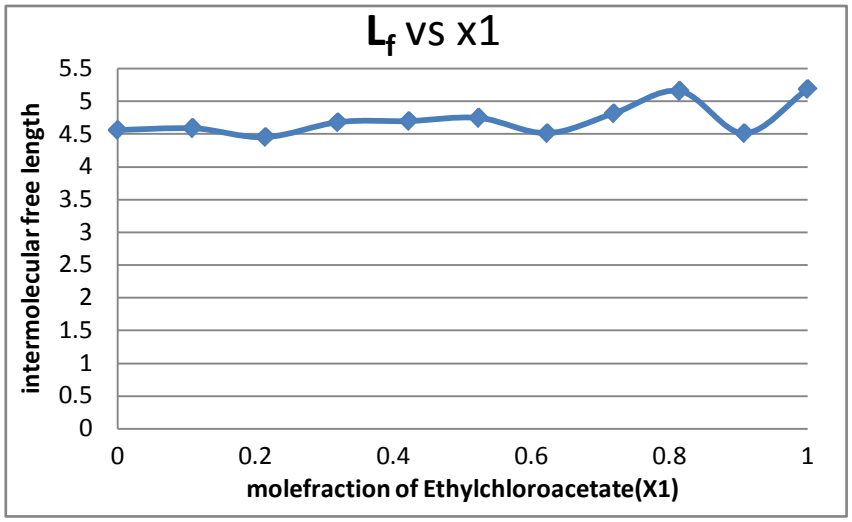


Figure 13 : Plot of mole fraction of ethylchloroacetate Vs Intermolecular free length

IV. Conclusion

Viscosities and densities for the binary liquid mixture of Ethylchloroacetate and Acetophenone system was found out as a function of mole fraction at atmospheric pressure and at temperatures of 303K and323K. From the density and viscosity data, the values of excess molar volumes (V^E) and the viscosity deviations ($\Delta\eta$) were determined at 303K and 323K. Excess molar volumes (V^E) and the viscosity deviations ($\Delta\eta$) were used to predict the intermolecular interactions in the mixtures. McAllister's three-body-interaction model, Krishnan-Laddha model and Jouyban-Acree model were used to correlate the kinematic viscosity of the systems. The excess volume and viscosity deviation data were fitted by means of the Redlich-kister equation. It was found that in all cases the experimental data obtained, matches with the McAllister model and Redlich-Kister equation with a high degree of precision.

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Table 6 : Predicted excess molar volume, isentropic compressibility (ks) , intermolecular free length(l_f) by (R-K model) for the mixture at 303 K

Molefraction of EthylChloroacetate	V^E(pred) (cc/gmole)	$\Delta\eta$ (pred)	ΔKs(pred)	ΔL_f (pred)
0	0	0	0	0
0.1089	-4.1232	-0.2409	4.7433	4.5181
0.2151	-3.2017	-0.2639	5.1736	4.0809
0.3199	-1.0267	-0.1541	4.3877	4.0530
0.4223	-1.7988	-0.1231	5.1930	4.9025
0.5231	-3.1453	-0.1376	5.5625	5.2154
0.6224	-2.036	-0.1086	4.8229	4.3045
0.7191	0.8659	-0.0444	4.9854	4.2894
0.8144	2.4135	-0.01189	6.3702	5.6870
0.9086	1.246	-0.0147	4.7013	4.3620
1	0	0	0	0

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Water Melon Seed as a Potential Coagulant for Water Treatment

By I.M. Muhammad, S. Abdulsalam, A. Abdulkarim & A.A. Bello
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Abstract- This paper reports the potential of watermelon seed as a natural coagulant for water treatment. It was aimed at identifying watermelon seed as a possible replacement for alum and other synthetic polyelectrolytes in treating water. Laboratory scale studies using jar test experiments were performed on medium turbid water to determine the effect of dosage, pH stirring time and speed on coagulation. Results obtained showed that at dosage of 0.1g/L, pH of 7.0, stirring time of 8 minutes and mixing speed of 100rpm, optimal removal of turbidity was obtained. The reduction in turbidity was below the world health organizations (WHO) recommended value of 5NTU, however the best colour removal was not up to the WHO recommended value of 40TCU. When used in combination with alum, it caused unfavourable changes in the pH of the treated water howeverwith 20% alum as coagulant aid, the best colour and turbidity removal at acceptable pH was obtained, with residual turbidity of 0.89 NTU and residual colour of 15TCU at a pH of 6.50. The results showed that watermelon seed can be used as a natural coagulant for water treatment.

Keywords: *water melon, seed, coagulant, turbidity, colour, water treatment.*

GJRE-C Classification : *FOR Code: 090410*

Strictly as per the compliance and regulations of :

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Keywords: water melon, seed, coagulant, turbidity, colour, water treatment.

I. Introduction

Water supply is a basic need required for living creatures and human being specifically. Developing countries and third world countries are facing potable water supply problems because of inadequate financial resources. The cost of water treatment is increasing and the quality of river water is not stable due to suspended and colloidal particle load caused by land development and high storm runoff during the rainy seasons. During the rainy seasons the turbidity level increases and the need for water treatment chemicals increase as well, which leads to high cost of treatment which the water treatment companies cannot sustain. As a result, the drinking water that reaches the consumer is not properly treated (Muyibi *et al.,* 2009). Therefore, it is of great importance to find a natural alternative for water coagulant to treat the turbidity. In this world the amount of resources available to living creatures are limited. Safe drinking water is essential to the health and welfare of a community, and water from all sources must have some form of purification before consumption (Arnoldsson *et al.,* 2008).

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Drinking water treatment involves a number of combined processes based on the quality of the water source such as turbidity, amount of microbial load present in water and the others include cost and availability of chemicals in achieving desired level of treatment (Muyibi *et al.,* 2009). Conventional methods used for purification of water include coagulation, sedimentation, filtration, aeration and also chemical treatment.

In drinking water treatment, the coagulation process is used to destabilize suspended particles and to react with organic materials in the raw water. Proper coagulation is essential for good filtration performance and for disinfection by product (DBP). Common coagulants are aluminium sulphate, ferric chloride, poly-aluminium chlorides and synthetic polymers. The use of coagulants such as alum is one of the commonest methods employed and it reduces the repulsive force between particulate matter, encouraging particle collision and floc formation (Moramudaii and Fernando, 2001).

Recent studies have indicated a number of serious drawbacks linked to the use of aluminium salts such as Alzheimer's disease associated with high aluminium residuals in treated water, excessive sludge production during water treatment and considerable changes in water chemistry due to reactions with the OH– and alkalinity of water. In addition, the use of alum salts is inappropriate in some developing countries because of the high costs of imported chemicals and low availability of chemical coagulants (Adejumo *et al.,* 2013). In addition, monomers of some synthetic organic polymers such as acrylamide have neurotoxicity and strong carcinogenic properties and because of this, there has been considerable interest in the development of natural coagulants which are safe for human health and biodegradable (Ghebremichael, 2004).

A number of studies have pointed out that the introduction of natural coagulants as a substitute for metal salts may ease the problems associated with chemical coagulants. Using natural coagulants instead of aluminium salts might give advantages, such as lower costs of water production, less sludge production and ready availability of reagents. There are also some disadvantages such as increased concentration of nutrients and chemical oxygen demand (COD) in the treated water due to the organic nature of this type of coagulants (Daniyan *et al.,* 2011).

Among plant materials that have been tested over the years, the seeds from *Moringa oleifera* have been shown to be one of the most effective primary coagulant in water treatment or purification. *Moringa oleifera* is the best natural coagulant discovered so far that can replace aluminium sulphate (alum), which is used widely for water treatment around the world (Ali *et al.,* 2010). Recently, however, there has been a resurgence of interest in natural coagulants for water treatment in developing countries. For this purpose the greatest degree of attention has been focused on the seed of Moringa oleifera from Sudan, Nirmali seed in India, mesquite bean and in Venezuela, red bean and common bean, sweet corn and so on. These natural coagulants can be used alone or as a substitution for chemical coagulants and flocculants. They can be used for reducing turbidity and microorganisms in water, for water softening and for dewatering sludge (Mirjana *et al.,* 2010).

Large populations in rural and semi-urban areas of Africa have no access to clean drinking water. Waterborne diseases, though a global health threat, is a feature of developing countries whose populace are compelled to use turbid and contaminated water for domestic purposes. The removal of colloidal and suspended particles present in water would be extremely beneficial as it would assuage the majority of problems associated with turbidity. Conventionally, removal of the colloids in water could be achieved by coagulation, using certain chemical coagulants like certified alum. For many developing countries, this treatment process is not feasible because of the high costs involved and the difficulty in assessing chemical coagulants including alum. Moreover, recent studies have pointed out the health threats arising from the consumption of residual aluminium present in water, such as Alzheimer's diseases and neurodegenerative illness.

This paper aimed at investigating the potential of water melon seed as a coagulant for water treatment. This material was selected because the watermelon seed has high protein content and some authors have considered that the active coagulant agents in plant extract are proteins. The objective of this study is to determine the potential of watermelon seed as a natural coagulant, and investigate the coagulation characteristics of its crude protein extracts.

II. Materials and Methods

a) Materials

Materials used in this work included watermelon seed cake (coagulant), N-hexane, Gubi Dam raw water in Bauchi-Nigeria, Distilled water, Soxhlet extractor, Digital pH meter, Electronic weighing balance, thermometer, drying oven, electric hot plate, flocculator, beakers, pipette, turbidimeter, conductivity meter, and stop watch (timer). All reagents used are of analytical grade.

b) Methods

i. *Water melon seed (coagulant) preparation*

Fresh seeds of watermelon *(Citrullus lanatus)* of the cucurbitaceae family were obtained from the local market *(Muda Lawal)* in Bauchi, Bauchi State, Nigeria. The fruits were sliced open using a clean stainless steel laboratory knife. The seeds were washed severally with water, sun-dried for a week, sorted to remove bad ones, shelled and ground with a high speed laboratory electric blender, packed in an air tight container. 150g of the crushed seeds were then packed in a thimble and placed in a soxhlet extraction apparatus. 500ml of the n-Hexane was used to extract oil from the crushed seed in the column. The apparatus was left running for about 6hours and stopped when the extraction was complete. The cake was then washed with distilled water to remove residual n-Hexane, dried in an oven till constant weight and then sieved. The finer particles were then used as the coagulant.

ii. *Sample water collection*

The raw water sample was collected from Gubi dam, located in Bauchi state, North-East of Nigeria. The water was collected from the side of river by immersing a plastic container until it was full. The cap was inserted while it was still underway. The water was then treated using the prepared coagulant.

iii. *Water quality tests*

a. Turbidity

Turbidity of the water sample was measured before and after treatment using a turbidimeter in accordance with the international method of water quality measurement and the results recorded.

b. Total Solid

Sample of the raw water was taken in 100ml beaker. A clean and dry crucible was weighted empty and the sample was then poured into it and reweighed. The respective weights were recorded and the crucible together with the sample water were then placed on a hot plate at 104oC to evaporate the water. When all the water evaporated, the crucible was allowed to cool down and reweighed together with the residue. The total solid present was then calculated using the equation:

$$TS = 100(A - B)/200ml$$ Where:

A = weight of (crucible + water) - weight of crucible empty

B = weight of (crucible + residue)

c. Total Suspended Solid (TSS)

Sample of the raw water was 100ml in a sample bottle. The weight of a dry filter paper was taken empty and the sample water was then filtered and the residue dried at 35-40oC in an oven. The new weight of the filter paper plus residue is then taken. The difference in the weight of the filter paper empty and with residue after drying was calculated and divided by total volume of sample.

d. Total Dissolved Solid (TDS)

This was obtained by taking the difference between TSS and TS or two-thirds of the conductivity using the conductivity meter.

e. pH

The pH of the samples was taken using an electronic pH meter.

f. Colour

Colour of the water sample was carried out before and after treatment using a turbidity meter.

g. Jar test

The jar test apparatus was used to carry out coagulation and flocculation on the water samples. Six 1litre beakers were used to study the effect of coagulant dosage on coagulation, the effect of pH on coagulation and the effect of stirring time and speed on coagulation. The following parameters were then measured on the filtrate after the coagulation was completed; turbidity, colour, flocs weight, TDS and conductivity. Six different weights of the coagulant were placed in each beaker, the first having 0.1g, and the remaining five varying from 0.1-0.6g at 0.1g interval in order to determine the optimum dosage. The raw water sample was then added to make up the 250ml mark and the jars were then placed in the jar test kit and the stirrers lowered into each. The stirring speed was set at 150rpm for rapid mixing for 2 minutes and 80rpm 8minutes for slow mixing. After this was completed the samples were allowed to settle and the flocs filtered using a filter paper and the parameters listed above were measured on the filtrate. From the results obtained the dosage with the best results in colour and turbidity removal was taken as the optimum.

The procedure above was used again, however a dose of 0.1g was maintained in all six beakers. The pH was varied from 6.0-8.5 by the addition of few drops of 1M NaOH into the beakers to make it alkaline. A few drops of 1M H2SO4 solution was also added in the first beaker to make it slightly acidic at 6.0. The same speed and stirring time was used as above and the parameters listed above were measured after the coagulation-flocculation and filtration process. The pH at which the best turbidity and colour removal were observed at was taken to be the optimum pH for coagulation.

Effect of coagulant dosage was also studied. The optimum dosage of 0.1g was used in all the beakers. The stirring speed was then varied ranging from 50rpm-300rpm at 50rpm interval. After the coagulation-flocculation process was completed for each, the samples were then filtered and the filtrate was used to test for the parameters. The same was done to determine the optimum stirring time, using the optimum speed to determine the best stirring time. The stirring time was varied at 2 mins, 5 mins, 8 mins, 10 mins and 15 mins for each beaker. After the coagulation-flocculation process was completed, the samples were then filtered and the filtrate was used for the tests (1-6).

III. Results and Discussions

The physicochemical properties of the raw water sample used in this study are presented in Table 1. From Table 1, the turbidity value of raw water was with the range of 50-150 NTU which is classified as medium turbidity water (Doerr, 2005).

Table 1 : Initial raw water properties

Parameter	Initial Result	WHO Standard
Temperature (^{0}C)	26.7	25-30
pH	6.82	6.5-8.5
Conductivity (μS/cm)	347	1400 Max.
Total dissolved solids (mg/L)	171	933 Max.
Turbidity (NTU)	63.5	5 Max.
Colour (TCU)	330	15 Max.

From Table 1, it can be seen that the turbidity and the colour are above the WHO's recommended value for good quality drinking water. Hence the need for treatment. However, all other components are within the accepted value and safe without treatment.

a) Effect of dosage on coagulation

Table 2 shows the results of the effect of coagulant dosage on coagulation. The dosages were varied from 0.1g/L - 0.6g/L for each sample treated. The settling time of 15 minutes was used and the samples filtered as longer time periods were observed for complete settling to take place.

Table 2 : Effect of coagulant dosage results

S/No	Dosage (g/L)	Temperature ^{0}C)	pH	Conductivity (μS/cm)	TDS (mg/L)	Turbidity (NTU)	Colour (TCU)
1	0.1	26.2	6.34	342	170	7.59	50
2	0.2	26.5	6.44	356	178	8.39	55
3	0.3	26.5	6.52	322	167	10.69	80
4	0.4	26.4	6.49	334	165	11.68	90
5	0.5	26.4	6.40	373	186	12.71	90
6	0.6	26.3	6.36	385	193	14.98	120

At varying coagulant dosages, the effect on constituent parameters is shown above in Table 2. At varying dosage no significant changes were observed on pH, temperature, conductivity and TDS for the water sample treated with watermelon seed cake as coagulant, however, there was a notable decrease in the turbidity of the water sample after treatment. The observation on pH and conductivity made in this present study were in accordance with previous studies on coagulation and flocculation ability of some seeds (Ndabigengesere et al., 1995). The greatest decrease was seen at the dose of 0.1g/L of raw water which reduced the turbidity from 63.5 to 7.58. This value is still above the WHO recommended level of 5NTU however according to Arnoldsson et al.,(2008), the optimal dosage for a specific water is defined as the dosage which gives the lowest turbidity in the treated water therefore the optimum dosage is 0.1g/L. At this dosage the efficiency of the coagulant in removing colour was also highest, reducing the colour from 330 to 50 TCU. This value is still large in comparison with the WHO recommended standard of less than 15TCU or PCU.

According to the findings of Ordonez et al. (2010) and Alo et al. (2012), it indicated that with increase of coagulant the conductivity increases however, this is finding is in agreement with their own as seen in Table 2 above.

b) Effect of pH on coagulation

Table 3 shows the results obtained when the pH of the raw water sample was varied to study the effect of pH on coagulation.

Table 3 : Effect of pH on coagulation results at dosage of 0.1g/L of watermelon seed cake

S/No	pH	Temperature (ºC)	Conductivity (µS/cm)	TDS (mg/L)	Turbidity (NTU)	Colour (TCU)
1	6.0	24.4	1726	863	5.28	40
2	6.5	24.7	1588	795	7.47	50
3	7.0	24.8	1660	831	4.23	45
4	7.5	24.6	351	177	5.13	40
5	8.0	24.7	406	203	4.69	45
6	8.5	24.6	413	206	5.76	55

In the coagulation–flocculation process, pH is very important since the coagulation occurs within a specific pH range for the coagulant (Othman et al., 2008). In this study a small range of pH, between 6.0 for slightly acidic medium and 8.5 for slightly alkaline medium was selected. Figure 1 shows the pattern followed by the effect of pH on water turbidity using watermelon seed coagulant.

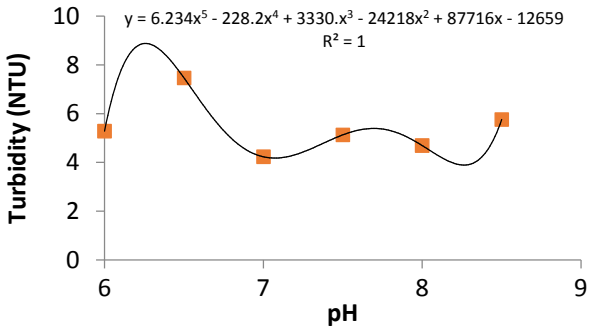

Figure 1 : plot of turbidity against pH showing the effect of pH on turbidity removal

It can be deduced from Fig. 1 that, variation of the water pH resulted in a 5 degree polynomial curve with R2 value of 1. pH of between 7-7.5 provides better response in turbidity.

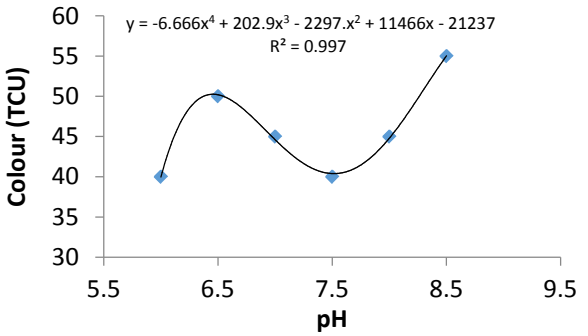

Figure 3 : Plot of colour against pH, showing the effect of pH on colour removal

According to Seyrig and Shan (2007), the probable reason why different pH provides different colour is that, the colour-producing substances in water behave inconsistently. pH adjustment may cause a change in the ionization of the colour molecule with corresponding effects on bond lengths and configurations and thus light absorption.

c) Effect of stirring time on coagulation at constant dosage

Table 4 shows the results obtained when the effect of stirring time on coagulation was studied by varying the stirring time at a constant coagulant dosage.

Table 4 : Effect of stirring time on coagulation at coagulant dosage of 0.1g/L

S/No	Time (min)	pH	Temperature (ºC)	Conductivity (µS/cm)	TDS (mg/L)	Turbidity (NTU)	Colour (TCU)
1	2.0	7.6	23.7	421	211	4.62	65
2	5.0	7.55	23.7	424	212	4.89	55
3	8.0	7.59	23.7	418	209	3.77	40
4	10.0	7.53	23.7	423	211	4.08	50
5	12.0	7.47	23.7	411	206	4.17	45
6	15.0	7.26	23.7	462	230	5.57	70

It can be seen from Table 4 that, the effect of stirring time on coagulation and as with the effect of dosage, the results obtained showed no significant changes in pH or temperature. The temperature remained constant, over a range of stirring time. Stirring time of 2-15min was used to show this. The TDS values obtained were still below 300 mg/L which are excellent, it has its highest value at the highest stirring time. Also at stirring time of 8min a turbidity value below the WHO recommended value was obtained, best colour removal of 40 TCU.

d) Effect of mixing speed on turbidity and colour

From Table 5, the effect of mixing speed on coagulation was observed to only have a moderate effect on the coagulation process. This is in accordance with the findings of Othman et al., (2008).

Table 5 : Effect of mixing speed on coagulation at dosage of 0.1g

S/No	Speed (rpm)	pH	Temperature (°C)	Conductivity (µS/cm)	TDS (mg/L)	Turbidity (NTU)	Colour (TCU)
1	50	7.19	26.4	387	194	5.27	55
2	100	7.32	26.2	403	201	5.23	50
3	150	7.42	26.1	404	202	5.57	60
4	200	7.45	26.3	418	209	6.17	60
5	250	7.51	26.3	368	184	7.90	75
6	300	7.58	26.4	433	216	6.01	60

In determining the effect of stirring speed on a range of 50-300rpm at an interval of 50 rpm was chosen. Slight changes were observed for the pH and temperature values, however the pH after coagulation remained more neutral than acidic or alkaline. There was no much difference in the temperature change from the original to that after coagulation; therefore there is no marked effect on temperature. The TDS and conductivity values show a steady increase in their trend however, at speed of 250rpm there was a sharp decline in turbidity with the trend following a polynomial law (Fig. 4).

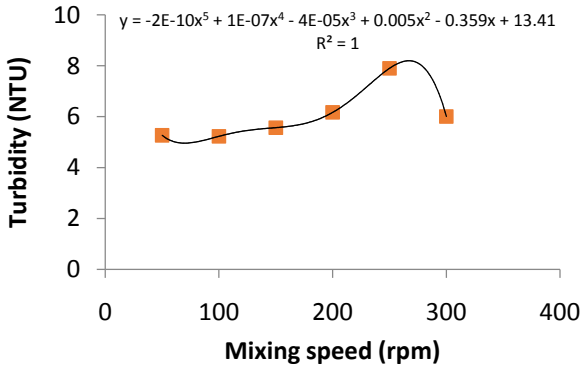

Figure 4 : Effect of mixing speed on turbidity removal

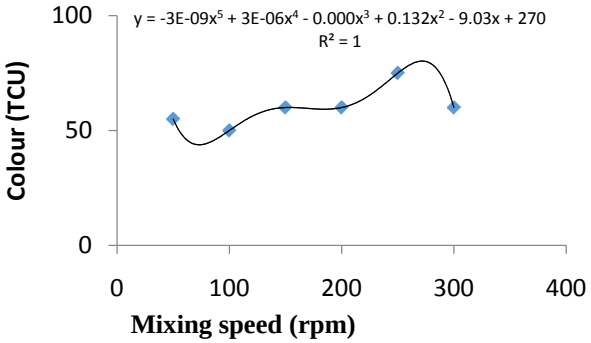

Figure 5 : Effect of mixing speed on colour removal

Also at 250rpm the turbidity and colour removal is least and best at stirring time of 100rpm with lowest turbidity value of 5.23 and colour value of 50TCU. No significant changes were recorded at speeds of 150 and 200rpm for both colour and turbidity. It can therefore mean that the lower mixing speed may improve the removal of turbidity due to reduced shearing of the flocs during initial formation which is in agreement with the findings of Ebeling et al (2004).

e) Effect of the combination of coagulant and coagulant aid on water treatment

Table 6 shows the results obtained after coagulation when alum was used as a coagulant aid and watermelon seed cake being used as the primary coagulant.

Table 6 : Effect of coagulant + coagulant aid on water treatment

S/No	Coagulant + Alum (Wt.%)	pH	Temperature (°C)	Conductivity (µS/cm)	TDS (mg/L)	Turbidity (NTU)	Colour (TCU)
1	100% C	7.26	25.5	419	211	6.79	20
2	80%C+20%A	6.50	25.4	472	236	0.89	15
3	60%C+40%A	6.09	25.6	478	238	0.47	5
4	40%C+60%A	4.63	25.5	564	284	1.04	5
5	20%C+80%A	4.46	25.6	646	322	1.04	0
6	100%A	4.21	25.6	873	437	0.95	5

Where:

A: Aluminium sulphate

C: Natural coagulant

The various coagulant combinations in Table 6 was used to treat the raw water sample (Gubi Dam raw water).

Results shown in Table 6, indicated significant changes in pH. With an initial raw water pH of 6.82, the sample with 100% watermelon cake, the pH recorded

was 7.26 which is neutral and well within the range of WHO standards. However steady increase in alum concentration showed a steady decline from neutral to acidic Fig. 6). This is in consonance with the results of Adejumo, et al., (2013) that showed that the pH of the water treated with natural coagulants (MO seed powder) was within the recommended WHO standards. At alum concentrations of only 40% the pH value had a significant shift towards acidic region and well within the recommended WHO standards. At concentrations of 60% and above, the pH was observed to become very acidic. This is also in consonance with the findings of Nwaiwu and Bello (2011) which showed that at alum of only 20% significant drop in pH was observed and at 80% the treated water was already acidic.

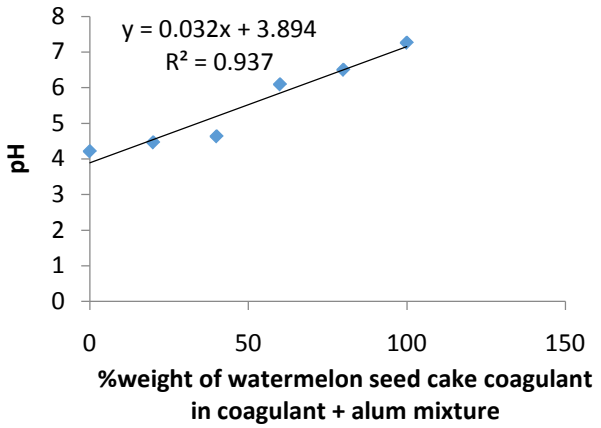

Figure 6 : Graph showing the effect of coagulant combination on pH of water

When the quantity of watermelon seed cake was more than the quantity of alum in any water sample treated, the pH reduction was within the WHO approved range of 6.0 to 8.5 but the reverse was the case when alum was either in equal quantity or greater amount than Watermelon seed cake in combination. In practical terms, this indicates that when using Watermelon seed cake alongside alum in water purification, the optimum combination should not necessitate further chemical addition for pH correction. This means that the quantity of alum in the combination should not exceed the quantity of Watermelon seed cake. The quantity of alum should be based on and should be a percentage of the Watermelon seed cake dosage suited for the water in question. Therefore a concentration of 20% alum is acceptable.

IV. Conclusion

From the study on the effects of pH, dosage, stirring speed and time, an optimum pH of 7.0, optimum dosage of 0.1g/L, optimum speed at 100rpm, and stirring time of 8 minutes were obtained respectively. Also, when watermelon seed cake was used in combination with alum higher colour and turbidity removal were observed, going as high as 100% clarification of colour. However the recommended ratio for the combined coagulant dose was 80% watermelon seed powder and 20% alum as best water treatment was obtained. This therefore establishes that watermelon seed powder as a natural coagulant can be more efficient when used with 20% alum as a coagulant aid. From the results obtained, watermelon seed has been found to be a potential natural coagulant for surface water treatment. Medium turbid water from Gubi dam was used as case study and a good efficiency of about 89% was obtained.

V. Recommendation

Based on the results obtained, the following recommendations were provided:

It is recommended that further research should be carried out on pilot scale water treatment using watermelon seed as coagulant and alum as coagulant aid.

It is also recommended that more research on highly turbid water as using the water melon seed powder as coagulant.

It is recommended that more natural sources should be investigated for potential coagulation abilities.

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Levenberg – Marquardt's Algorithm used for PID Controller Parameters Optimization

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Abstract- The determination of parameters of controllers is an important problem in automatic control systems. In this paper, the Levenberg Marquardt (LM) Algorithm is used to effectively solve this problem with reasonable computational effort. The Levenberg Marquardt (LM) Algorithm for optimization of three term (PID) controller parameters with dynamic model of pH neutralization process is presented. The main goal is to show the merits of Levenberg Marquardt algorithm optimization and to determine its suitability in the area of control systems. Lastly, the application of this approach to the calculation of the parameters of PID controller shows that the Levenberg Marquardt (LM) algorithm has a better dynamic performance of pH neutralization process.

Keywords: parameters optimization, PID controller, pH neutralization process, levenberg marquardt (LM) algorithm.

GJRE-C Classification : FOR Code: 090499

Strictly as per the compliance and regulations of :

Levenberg – Marquardt's Algorithm used for PID Controller Parameters Optimization

Ahmed S. Abd El-Hamid [α], Ahmed H. Eissa [σ] & ALy Radwan [ρ]

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I. Introduction

In mathematics and computing, the Levenberg–Marquardt Algorithm (LMA), also known as the damped least-squares (DLS) method, is used to solve non-linear least squares problems. These minimization problems arise especially in least squares curve fitting. The LMA interpolates between the Gauss–Newton Algorithm (GNA) and the method of gradient descent.

The LMA is more robust than the GNA, which means that in many cases it finds a solution even if it starts very far off the final minimum. For well-behaved functions and reasonable starting parameters, the LMA tends to be a bit slower than the GNA. LMA can also be viewed as Gauss–Newton using a trust region approach. The LMA is a very popular curve-fitting algorithm used in many software applications for solving generic curve-fitting problems. However, as for many fitting algorithms, the LMA finds only a local minimum, which is not necessarily the global minimum [1]. The primary application of the Levenberg–Marquardt algorithm is in the least squares curve fitting problem: given a set of m empirical datum pairs of independent and dependent variables, (xi, yi) optimize the parameters β of the model curve $f(x, \beta)$ so that the sum of the squares of the deviations becomes minimal.

$$S(\beta) = \sum_{i=1}^{m} [y_i - f(x_i, \beta)]^2$$

The Levenberg-Marquardt (LM) algorithm is an iterative technique that locates the minimum of a multivariate function that is expressed as the sum of squares of non-linear real-valued functions [4, 6].

In the other hand, a common problem in control system design is establishing the appropriate value of controller gains. In general a low value of gain produces a slow system response, while high gain values can cause an excessively-oscillatory response with the possibility of instability. Somewhere between these extremes is a value of gain that produces the best system response. The essential function of a feedback control system is to reduce the error, $e(t)$ between any variable and its demanded value to zero as quickly as possible. Therefore, any criterion used to measure the quality of system response must take into account the variation of the error over the whole range of time. Four basic criteria are in common use:

Integral of absolute error (IAE) $= \int_0^\infty |e(t)|dt$

Integral of squared error (ISE) $= \int_0^\infty \{e(t)\}^2 \, dt$

Integral of time multiplied by absolute error (ITAE) $= \int_0^\infty t \, |e(t)|dt$

Integral of time multiplied by squared error (ISE) $= \int_0^\infty t \{e(t)\}^2 dt$

For any of the possible criteria, the best response corresponds to the minimum value of the chosen criterion. Note that in all cases it is either the absolute error or the squared error which is involved, straightforward integration of the error would produce zero result, even if the system response was a constant amplitude oscillation. IAE is often used where digital simulation of a system is being employed, but it is inapplicable for analytical work, because the absolute value of an error function is not generally analytic in form. This problem is overcome by the ISE criterion. The ITAE and ITSE have an additional time multiplier of the error function, which emphasizes long-duration errors, and therefore these criteria are most often applied in systems requiring a fast settling time [3, 5].

In this paper, the shape of the complete closed loop response, from time $t = 0$, until steady state has been reached, could be used for the formulation of a

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dynamic performance criterion. The simple criteria of this category are based on the entire response of the process and the integral of the Square Error (ISE) criterion used here, where

$$ISE = \int_0^\infty e^2(t)\,dt = \int_0^\infty \{Y_{sp}(t) - Y(t)\}^2\,dt \qquad (1)$$

Where $e(t) = Y_{sp}(t) - Y(t)$ is the deviation (error) of the response from the desired set point.

The ideal continuous time domain PID controller for a SISO process is expressed in the Laplace domain as follows:

$$G(s) = K_p + \frac{K_i}{s} + K_d S \qquad (2)$$

With K_p = proportional gain, K_i = integral time constant and K_d = derivative time constant.

II. Mathematical Model of pH Neutralization Process

Consider a pH neutralization process as shown in Fig. 1. The flow rates of acid, buffer, base and effluent streams are denoted by q_1, q_2, q_3, and q_4, respectively. Output of the process is the pH value of the effluent stream, and the flow rate of base stream, q_3 is the control input. A dynamic model is derived using the conservation laws and reactions equilibrium. The modeling assumptions include perfect mixing, constant volume of the neutralization tank (V), and complete solubility of the ions involved. The chemical reactions in the system are as follows [8]:

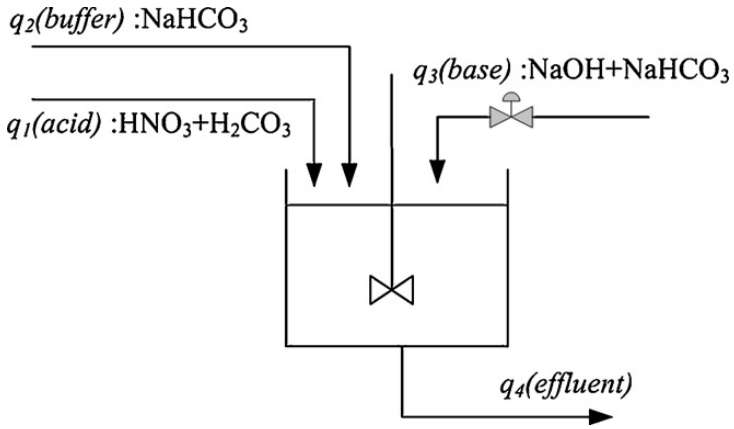

Fig. 1 : pH neutralization process

$$H_2CO_3 \leftrightarrow H^+ + HCO_3^-$$
$$HCO_3^- \leftrightarrow H^+ + CO_3^{2-}$$
$$HNO_3 \rightarrow H^+ + NO_3^-$$
$$NaHCO_3 \rightarrow Na^+ + HCO_3^-$$
$$NaOH \rightarrow Na^+ + OH^-$$

The equilibrium constants for these reactions are:

$$Ka_1 = \frac{[HCO_3^-][H^+]}{[H_2CO_3]}$$

$$Ka_2 = \frac{[CO_3^{2-}][H^+]}{[HCO_3^-]}$$

$$Kw = [H^+] + [OH^-] + [CO_3^{2-}]$$

The chemical equilibrium equations are modeled using the reaction invariant concept. For this system, concentrations of reaction invariants are defined as:

$$x_1 = [NO_3^-]$$
$$x_2 = [Na^+]$$
$$x_3 = [H_2CO_3] + [HCO_3^-] + [CO_3^{2-}]$$

Denoting, $y = pH$ the ions neutrality balance in the tank results the following static equation:

$$h(x,y) = -x_1 + x_2 + x_3 c_{x3} + 10^{-y} - 10^{y-PK_w} = 0 \qquad (3)$$

$$c_{x3} = \frac{2 + 10^{PK_2 - y}}{1 + 10^{PK_2 - y} + 10^{PK_1 + PK_2 - 2y}}$$

$$PK_1 = -\log_{10} Ka_1$$

$$PK_2 = -\log_{10} Ka_2$$

The dynamic equations are given by:

$$\frac{d\dot{x}_1}{dt} = \frac{q_1}{V}(w_{11} - x_1) + \frac{q_2}{V}(w_{21} - x_1) + \frac{q_3}{V}(\propto_1 - x_1) \qquad (4)$$

$$\frac{d\dot{x}_2}{dt} = \frac{q_1}{V}(w_{12} - x_2) + \frac{q_2}{V}(w_{22} - x_2) + \frac{q_3}{V}(\propto_2 - x_2) \qquad (5)$$

$$\frac{d\dot{x}_3}{dt} = \frac{q_1}{V}(w_{13} - x_3) + \frac{q_2}{V}(w_{23} - x_3) + \frac{q_3}{V}(\propto_3 - x_3) \qquad (6)$$

Where:

V: Volume of the mixing tank, ml

Kw: Dissociation constant of water, 10^{-14}

Ka_i: ith dissociation constant of acid

w_i: Concentration of the ith species in the process stream, mol/l

w_{1i}: Concentration of the ith species in the acid stream, mol/l

w_{2i}: Concentration of the ith species in the buffer stream, mol/l

q_i: Flow rate of acid, buffer and base stream in simulation, ml/s

$\propto_i$: Concentration of the ith species in the titrating stream, mol/l

x_i: Reaction invariant of ith species, mol/l

y: Process variable, pH

u: Flow rate of the titrating stream, ml/min or ml/s

III. Simulation Results

The closed loop control system was solved using Levenberg–Marquardt's optimization approach with sampling time of 0.001 s. The simulation method combines SIMULINK module for pH neutralization model and M-file for LMA approach. A list of M-file programs used in the paper is provided in Appendix 1 and 2. Figure (1 and 2) show the responses of the pH neutralization obtained with change in pH set point. The optimal gains of PID controller are calculated to minimize the error function which described in equation (2). Also the values of gains of PID controller are plotted. The response of pH value tends to set point value with minimum steady state error and the values of gains tend to minimum values once the error reaches to zero value. Figure (3) show the change in set point from pH = 6 to 7, noticed that the spike value of manipulated variable (q_3) and the values of gains (Kp and Ki) tend to increasing while the gain (Kd) tends to decreasing. In other hand, figure (4) show the change in set point from 7 to 6, the value of manipulated variable (q_3) and the value of gains (Kp, Ki and Kd) tend to decreasing.

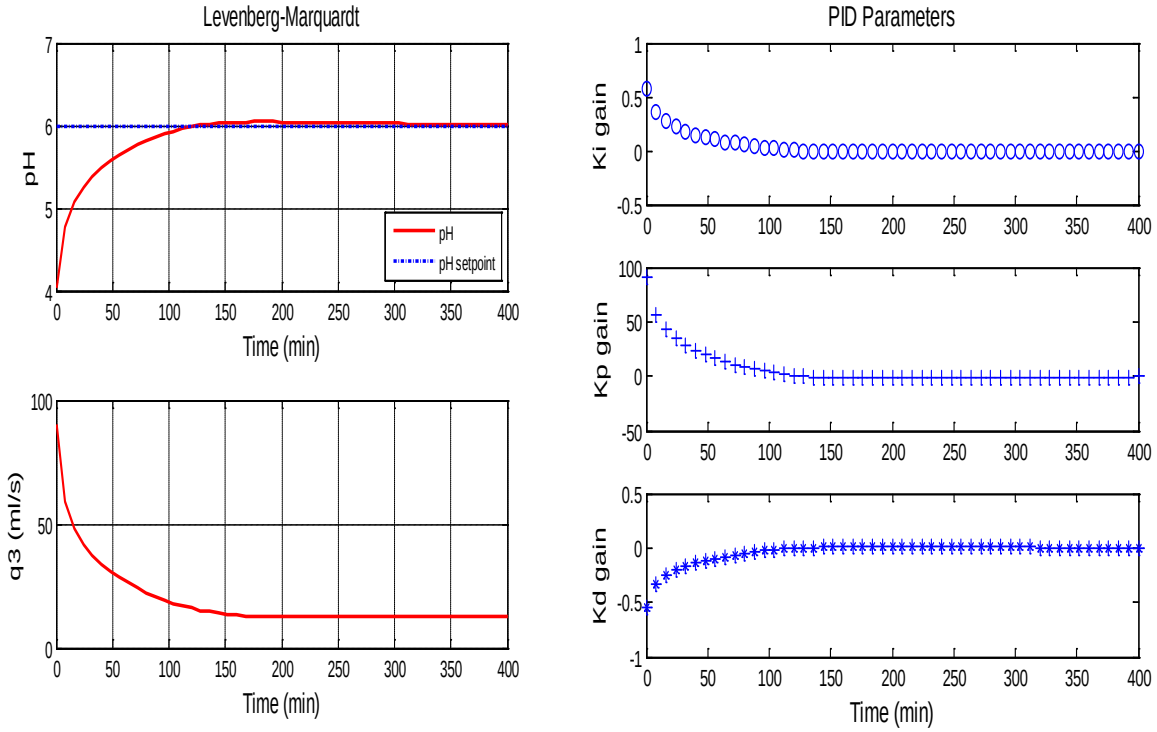

Fig. 1 : Simulation results of PID controller and values of parameters

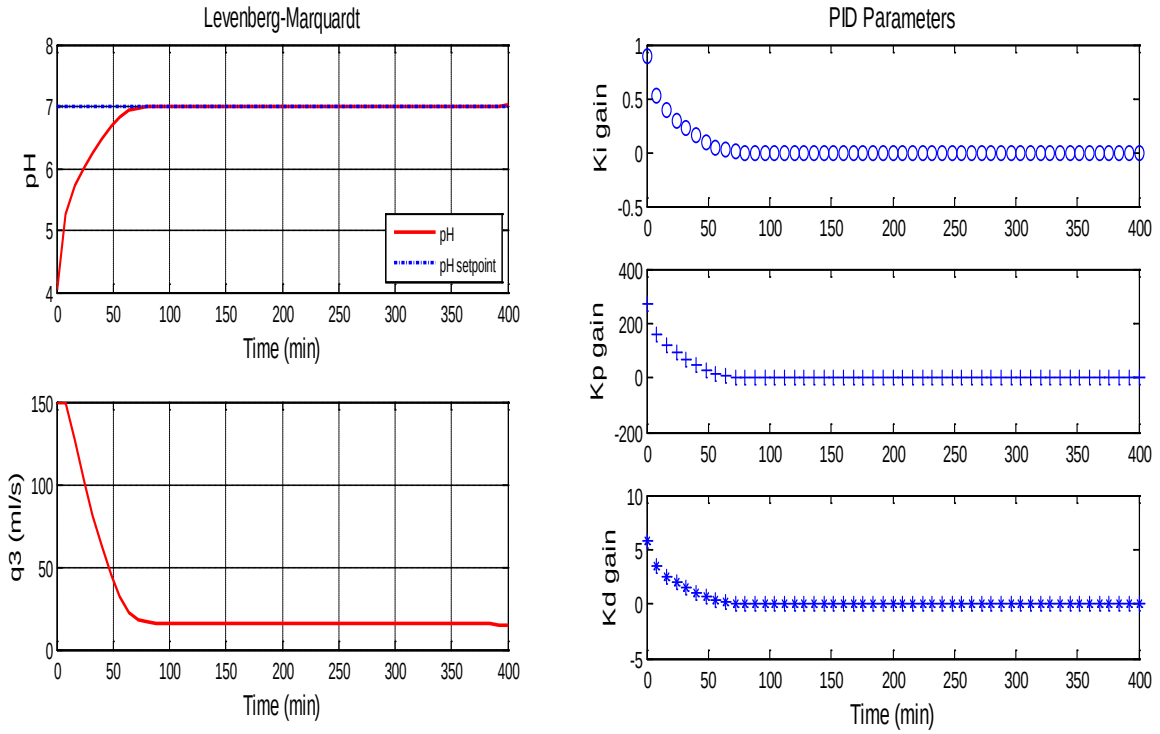

Fig. 2 : Simulation results of PID controller and values of parameters

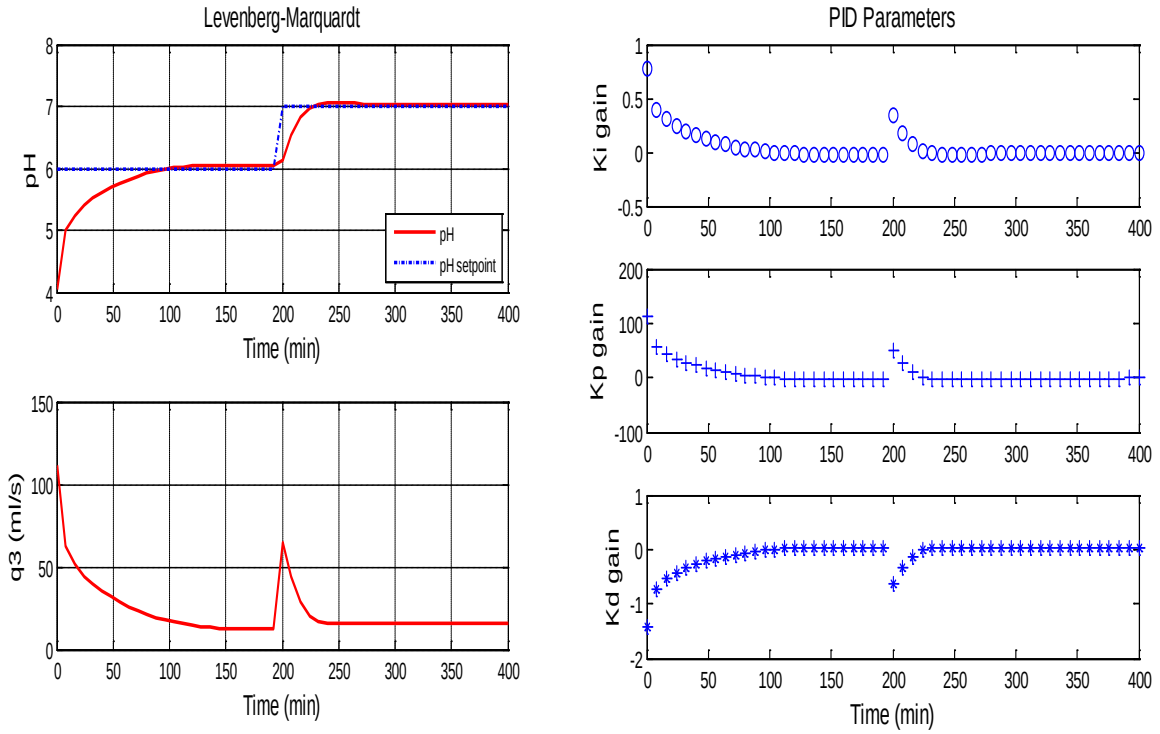

Fig. 3 : Simulation results of PID controller and values of parameters

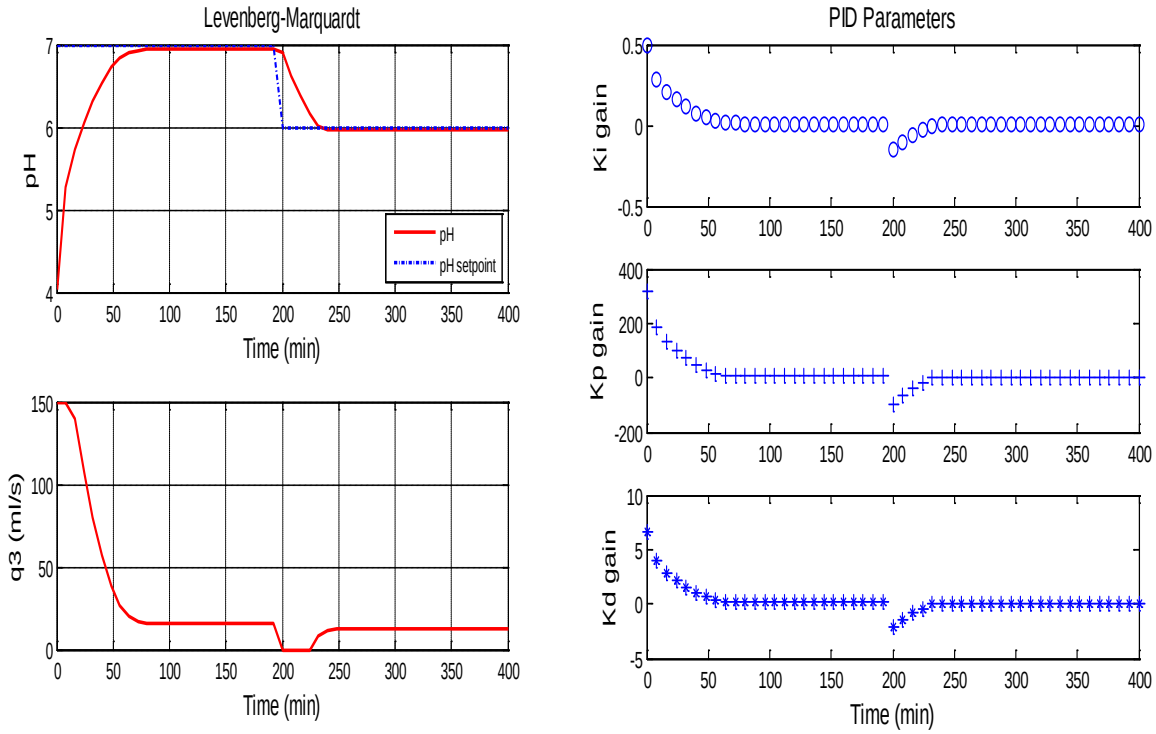

Fig. 4 : Simulation results of PID controller and values of parameters

IV. Conclusion

The paper presents an application of the Levenberg-Marquardt Algorithm (LMA) to optimization of parameters (Kp, Ki, Kd) of the PID controller structure according to minimum of integral square error. The simulated results were obtained of parameters by means of computer program implemented in Matlab software. As an example, the optimization of parameters of PID controllers with reference to a ph Neutralization process was presented.

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Appendix (1)

```
%Determination of PID controller prameters using "Levenberg-Marquardt %Algorithm"
%Step 1: Write an M-file tracklsq.m.
function [Kp,Ki,Kd] = phpidL2015k
ph2015 % Load the simulink model
```

```
k0 = [0 0 0]; % Set initial values of parametrs
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%%%Create or edit optimization options structure
options = optimset('Algorithm','levenberg-marquardt','Display','iter',...
'TolX',1e-5,'TolFun',1e-9,'TolCon',1e-6);
k = lsqnonlin(@tracklsq, k0, [], [], options);
Kp = k(1); Ki = k(2);Kd = k(3);
function F = tracklsq(k)
Kp = k(1);
Ki = k(2);
Kd = k(3);
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% Choose solver and set model workspace to this function
%Step 2: Invoke optimization routine.
simopt = simset('solver','ode5','SrcWorkspace','Current');
[t,x,y1,y2,y3] = sim('ph2015',[0 400],simopt);

%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%F = set point - actual value; % Compute error value
F = y2-y1;
end
%
% Put variables back in the base workspace
Kp = k(1)
Ki = k(2)
Kd = k(3)
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%plot(tout,yout,'r',tout,yset,'-b');
% Set axes and labels.
%axis([0 30 -1.6 1.6]); xlabel('Time'); ylabel('Amplitude');
clf reset
 subplot(2,1,1),plot(t,y1,'r',t,y2,'-.b','LineWidth',2);
 title('levenberg-marquardt')
 legend({'pH','pH setpoint'},'Location','SE','FontSize',8')
 xlabel('Time (min)','FontSize',12')
 ylabel('pH','FontSize',12)
 grid on

 subplot(2,1,2),plot(t,y3,'r','LineWidth',2);
 xlabel('Time (min)','FontSize',12')
 ylabel('q3 (ml/s)','FontSize',12')

 grid on
 end
```

Appendix (2)

```
% Read Mat. Files of PID gains
% ki(First number, second number)first number points to raw number
% second number points to col. number
%
load ki.mat; %# assume this contains a matrix called ki
load kp.mat
load kd.mat
for i=1:1:51 %# numbers of columns
x(i) = ki(1,i); %# numbers from all rows, column 1, into X
x1(i)=kp(1,i);
x2(i)=kd(1,i);
y(i)= ki(2,i); %# numbers from all rows, column 2, into Y
y1(i)= kp(2,i);
y2(i)=kd(2,i);

subplot(3,1,1),(plot(x,y,'o'));
title('PID Parameters','FontSize',12')
ylabel('Ki','FontSize',12)
subplot(3,1,2),(plot(x1,y1,'+'));
ylabel('Kp','FontSize',12)
subplot(3,1,3),(plot(x2,y2,'*'));
xlabel('Time (min)','FontSize',12')
ylabel('Kd','FontSize',12)

end
```

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Content

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- In manuscript, explain each of your consequences, point the reader to remarks that are most appropriate.
- Present a background, such as by describing the question that was addressed by creation an exacting study.
- Explain results of control experiments and comprise remarks that are not accessible in a prescribed figure or table, if appropriate.
- Examine your data, then prepare the analyzed (transformed) data in the form of a figure (graph), table, or in manuscript form.

What to stay away from

- Do not discuss or infer your outcome, report surroundings information, or try to explain anything.
- Not at all, take in raw data or intermediate calculations in a research manuscript.
- Do not present the similar data more than once.
- Manuscript should complement any figures or tables, not duplicate the identical information.
- Never confuse figures with tables - there is a difference.

Approach

- As forever, use past tense when you submit to your results, and put the whole thing in a reasonable order.
- Put figures and tables, appropriately numbered, in order at the end of the report
- If you desire, you may place your figures and tables properly within the text of your results part.

Figures and tables

- If you put figures and tables at the end of the details, make certain that they are visibly distinguished from any attach appendix materials, such as raw facts
- Despite of position, each figure must be numbered one after the other and complete with subtitle
- In spite of position, each table must be titled, numbered one after the other and complete with heading
- All figure and table must be adequately complete that it could situate on its own, divide from text

Discussion:

The Discussion is expected the trickiest segment to write and describe. A lot of papers submitted for journal are discarded based on problems with the Discussion. There is no head of state for how long a argument should be. Position your understanding of the outcome visibly to lead the reviewer through your conclusions, and then finish the paper with a summing up of the implication of the study. The purpose here is to offer an understanding of your results and hold up for all of your conclusions, using facts from your research and generally accepted information, if suitable. The implication of result should be visibly described. Infer your data in the conversation in suitable depth. This means that when you clarify an observable fact you must explain mechanisms that may account for the observation. If your results vary from your prospect, make clear why that may have happened. If your results agree, then explain the theory that the proof supported. It is never suitable to just state that the data approved with prospect, and let it drop at that.

- Make a decision if each premise is supported, discarded, or if you cannot make a conclusion with assurance. Do not just dismiss a study or part of a study as "uncertain."
- Research papers are not acknowledged if the work is imperfect. Draw what conclusions you can based upon the results that you have, and take care of the study as a finished work
- You may propose future guidelines, such as how the experiment might be personalized to accomplish a new idea.
- Give details all of your remarks as much as possible, focus on mechanisms.
- Make a decision if the tentative design sufficiently addressed the theory, and whether or not it was correctly restricted.
- Try to present substitute explanations if sensible alternatives be present.
- One research will not counter an overall question, so maintain the large picture in mind, where do you go next? The best studies unlock new avenues of study. What questions remain?
- Recommendations for detailed papers will offer supplementary suggestions.

Approach:

- When you refer to information, differentiate data generated by your own studies from available information
- Submit to work done by specific persons (including you) in past tense.
- Submit to generally acknowledged facts and main beliefs in present tense.

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Methods and Procedures	Clear and to the point with well arranged paragraph, precision and accuracy of facts and figures, well organized subheads	Difficult to comprehend with embarrassed text, too much explanation but completed	Incorrect and unorganized structure with hazy meaning
Result	Well organized, Clear and specific, Correct units with precision, correct data, well structuring of paragraph, no grammar and spelling mistake	Complete and embarrassed text, difficult to comprehend	Irregular format with wrong facts and figures
Discussion	Well organized, meaningful specification, sound conclusion, logical and concise explanation, highly structured paragraph reference cited	Wordy, unclear conclusion, spurious	Conclusion is not cited, unorganized, difficult to comprehend
References	Complete and correct format, well organized	Beside the point, Incomplete	Wrong format and structuring

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