

Review of Solvation Studies of Copper (I) Complexes in Different Solvents and at Different Temperatures

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DOI: <https://doi.org/10.52403/ijrr.20240211>

ABSTRACT

The study of the solvation behavior of copper (I) complexes in the mixed solvents has generated a great interest in the field of chemistry. Copper complexes have attracted wide attention due to their important role in analytical chemistry, organic synthesis, metallurgy and medicines etc. Nevertheless, during recent years there has been an increasing interest in the behavior of copper (I) complexes in aqueous and non-aqueous solvents with a view to investigate ion-ion and ion solvent or solute-solute interactions under varied conditions.

The solvation behavior of some copper(I) perchlorate complexes in binary mixtures of dimethylsulfoxide with dimethylacetamide and dimethylformamide at 298K, and Some Copper(I) Nitrate Complexes in Dimethylsulfoxide and Nitromethane at 298 K and copper sulfate have been measured in binary mixtures of aqueous-methanol mixtures at different temperatures is studied by a combined approach using Molar conductance, ultrasonic velocity and density data, or limiting molar conductance (Λ_0), the association constants (KA) respectively. This study is a condensed version of earlier work in the same fields.

Keywords- solvation, Molar conductance, Copper sulfate, copper(I) perchlorate

INTRODUCTION

Solvation is the interaction of a solvent with the dissolved solute—in the case of water, solvation is often referred to as *hydration*. Solvent polarity is the most important factor

in determining how well it solvates a particular solute.

Solvation chemistry involves the analysis of interactions in the solution by the solute and the solvent. It is of fundamental importance since it provides relevant information regarding the nature of solvents and the extent of interaction among them. One of the important features of solvation studies is the deduction of the exact molecular structure of the solvent in a solution, with accuracy. Because, once an ion or solute is introduced in the solvent, it modifies the latter's structure, and at the same time, the structure of the solute is also changed. Thus, the solvent-solvent, solute-solute, and solute-solvent interactions result in ionic solvation. Conductance and viscosity measurements provide sufficient information regarding solvation behavior of ions in pure and mixed solvents. Studying the information of the transport properties (conductance, viscosity, ionic mobility) of electrolytes in aqueous and partially aqueous media tell us all about ion-ion and ion-solvent interactions in these solutions.

Strong ion-ion interactions are dominant in concentrated solutions. The influence of ion-dipole interactions between solvent molecules and solute ions increases with dilution. In extremely dilute solutions, a predominant weak dipole-dipole interaction is present.

The research of copper complexes characteristics has recently become crucial in

a variety of industries and professions, including biochemistry. Copper perchlorate hexahydrate is used as an efficient catalyst for the synthesis of polyhydroquinolines. Copper sulfate can be used as a decorative, since it adds colour to cement. The pentahydrate of this compound, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ is used as a fungicide due to its ability to kill several fungi. Copper nitrate can be employed as an oxidant, catalyst, used in medicines as an insecticide, or nitration reagent.

Current interest in Cu complexes is stemming from their potential use as antimicrobial, antiviral, anti-inflammatory, antitumor agents, enzyme inhibitors, or chemical nucleases.¹

Studies of electrolyte solutions in mixed solvents have a wide range of industrial applications. The stabilized copper salt solutions have been used as catalysts in many oxidation-reduction reactions. Though solvation studies of Cu(I) salts were attempted in the recent years, however no attempt has been made to study the solvation behavior of monovalent copper nitrate complexes in binary mixtures of dimethylsulfoxide (DMSO) and nitromethane (NM).

The ultrasonic velocities of solutions of Bu_4NBPh_4 , Bu_4NClO_4 , $[\text{Cu}(\text{AN})_4]\text{NO}_3$, $[\text{Cu}(\text{BN})_4]\text{NO}_3$, $[\text{Cu}(\text{Phen})_2]\text{NO}_3$, $[\text{Cu}(\text{DMPPhen})_2]\text{NO}_3$, $[\text{Cu}(\text{Bipy})_2]\text{NO}_3$ and $[\text{Cu}(\text{TU})_4]\text{NO}_3$ have been measured in the concentration range 0.03-0.27 M in dimethylsulfoxide (DMSO), nitromethane (NM) and binary mixtures of DMSO + NM containing 0, 20, 40, 60, 80 and 100 mol% NM at 298 K in the present studies

Highly ionic copper(I) complexes like copper(I) perchlorate tetra acetonitrile ($\text{CuClO}_4 \cdot 4\text{AN}$) and copper(I) perchlorate tetra benzonitrile ($\text{CuClO}_4 \cdot 4\text{BN}$) are unstable in water and a large number of organic solvents. These copper(I) complexes, however, have a marked stability in acetonitrile. Therefore, studies of such salts in pure and mixed solvents are still lacking. These copper(I) complexes, however, have a marked stability in acetonitrile (AN),

benzonitrile (BN), and some other nitriles. Some investigations of the behavior of these white-coloured complexes have been made in pure acetonitrile and binary mixtures of acetonitrile with several other organic solvents.

In continuation with our studies of the copper(I) complexes in solution, in the present paper, we report the molar conductances of some copper(I) perchlorate complexes in binary mixtures of DMSO with DMA and DMF so that their solution behavior can be investigated thoroughly. DMSO, DMA and DMF were selected as solvents because copper(I) complexes had marked stability in these solvents.

The concept of ion association is widely used in solution chemistry. A literature survey shows that ion-pair or multiple-ion association phenomena and the nature of the solute-solvent interactions (in aqueous, non-aqueous and mixed solutions) have been studied conductometrically.

Fuoss-Shedlovsky conductivity equation has been successfully used by many researchers for the investigation of many electrolytes in solutions in which ionic association predominates. Alcohol-water mixtures at different temperatures exhibit a wide range of dielectric constants, viscosity and a high degree of hydrogen bonding effect, so that the present article aims to study the effect of dielectric constants, viscosity and the hydrogen bonding on the transport properties of copper sulfate in binary mixtures methanol-water ($\text{MeOH-H}_2\text{O}$) with the alcohol mass fractions of 0%, 20% and 40% at different temperatures (298.15, 303.15, 308.15 and 313.15) K by applying the Fuoss Shedlovsky conductivity equation.

2. MATERIAL AND SOLUTIONS

Copper (I) ion-

It is known that copper(I) ion has a distorted octahedral structure with an elongated axis in water, and the Cu—OH₂ distances are 194 pm within the plane and 243 pm along the axis. According to calorimetric studies on solvation of copper(I) ion with DMF in DMF-AN mixtures, solvated complexes

[Cu(dmf)_n]⁺ (n=1—4,6) forms the formation of Cu(dmf)₅⁺ has not being confirmed

Solutions –

DMSO (99.8%, E. Merck, India) and NM (99%, Loba Chemie, India) were purified according to the reported methods. Tetrabutylammonium tetraphenylborate [Bu₄NBPh₄] and tetrabutylammonium perchlorate [Bu₄NClO₄] used as reference electrolytes were prepared and dried.

Tetraacetonitrile copper(I) nitrate [Cu(AN)₄]⁺NO₃⁻, was synthesized by reduction of copper(II) nitrate trihydrate [Cu(NO₃)₂·3H₂O] (99%, Merck) (5 g) with copper powder (99.7% from Lobachem) (3 g) in warm acetonitrile (40 mL) and heated for 10-20 min till the solution became colourless at 60-70 °C. The colourless solution was filtered and on cooling white crystals separate. Crystals were dried and stored under vacuum. The synthesized tetraacetonitrile copper(I) nitrate crystals remained stable only for a short time and became blue in a few hours. However, copper(I) nitrate solution in dry acetonitrile remained stable and was used to synthesize other complexes with stronger ligands like phenanthroline, 2,9-dimethyl-1,10-phenanthroline (neocuproin), thiourea, bipyridyl.

Acetonitrile (AN) (99.8 %), benzonitrile (BN) (99.8 %), dimethylformamide (DMF) (99.5 %), dimethylacetamide (DMA) (99.5 %) and dimethylsulfoxide (DMSO) (99.5 %), all from E. Merck, were purified by the methods as reported. Tetrabutylammonium tetraphenylborate (Bu₄NBPh₄) and tetrabutylammonium perchlorate (Bu₄NClO₄), which were used as reference electrolytes in the present work were prepared by the methods already reported.

Copper(I) perchlorate tetraacetonitrile (CuClO₄·4AN) was prepared by the reduction of copper (II) perchlorate hexahydrate Cu(ClO₄)₂·6H₂O by copper powder in warm acetonitrile (AN) 99.8 % (E. Merck) following the method reported by Gill and Cheema.

Copper(I) perchlorate tetrabenzonitrile (CuClO₄·4BN) was prepared from Cu(ClO₄)₂·6H₂O by reduction with copper powder in benzonitrile (BN). Cu(ClO₄)₂·6H₂O was dissolved in anhydrous benzonitrile and the solution was heated at 70-80 °C.

Copper sulfate pentahydrate (CuSO₄·5H₂O, 99.5%), methanol (MeOH, 99%), all were supplied from Riedel-de Haën company (Germany) and used without further purification. Binary mixtures of methanol-water with the alcohol mass fractions of 0%, 20%, and 40% were prepared by applying the following equation:

$$\text{Alcohol percentage} = \frac{v_1 d_1 (100)}{v_1 d_1 + v_2 d_2}$$

where d₁ and d₂ are the density of alcohol and water respectively. V₁ is the volume of alcohol which will be added to the volume V₂ of water to get the mixture of the required percentage.

REVIEW OF LITERATURE

James G. Speight in his work Natural Water Remediation deals with the way in which water molecules are strongly Re attracted to most mineral surfaces. The effectiveness of water as a solvent is related to such activities. Its effectiveness in weathering rocks is also increased by the ability of this cohesive liquid to wet mineral surfaces and penetrate into small openings. [1]

Vivek pathania studied Transport Behavior of Copper(I) Nitrate Complexes in Pure Dimethylsulfoxide, Pyridine, Nitromethane and their Binaries Probed by Conductometer Parameters at 298 K by conductance measurements showed that. ion solvation increases in the order Py > DMSO > NM indicating stronger solvation in the case of Py than for DMSO and then for NM. [2]

Dip Singh Gill reported studies about Preferential solvation of some copper (I), silver (I) and sodium (I) salts in acetonitrile +n-butyronitrile and acetonitrile +N, N-dimethylacetamide mixture according to which Better insight into the solvation behaviour is provided by ionic viscosity B coefficients concluded that Cu⁺ is more

solvated in AN + DMA mixtures as compared to that in AN + nBTN. [3]

Veeneta in her Solvation Studies Of Copper (I) Sulphates In Non Aqueous Solvent Mixtures studies deduced theoretically ion-ion and ion-solvent or solute-solvent interactions under varied conditions.

According to Veeneta who took into account The salvation of copper(I) sulfate in binary mixtures of water and N,N-dimethyl formamide (DMF) by using electrochemical studies in solution and a mass spectrometric assay of the solvated ions formed from these solutions upon (ESI) [4]

Esam A. Gomaa at el. Studied Conductometric and volumetric study of copper sulphate in aqueous ethanol solutions at different temperatures in which essam A, Gomma measure the densities of copper sulphate solutions in H₂O and EtOH–H₂O at 298.15 K, 303.15 K, 308.15 K, and 313.15 K.

From the obtained data, the limiting molar conductance λ_m° , association constant K_A , energy of activation of the rating process (E_a), and related thermodynamic parameters were determined showing that difference of the volumetric properties reflects the significant influence of solvent and temperature on the salt behavior and thus the ion-pairing depends on the dielectric constant and different properties of the medium. The association decreases as the temperature increases and increases as the proportion of organic solvent increases. [5]

Isidoros Iakovidis in his study v Copper and Its Complexes in Medicine: A Biochemical Approach explains the fundamental role of copper and the recognition of its complexes as important bioactive compounds in vitro and in vivo as potential drugs for therapeutic intervention in various diseases. In spite of its great importance copper is potentially toxic because it may penetrate the blood/brain barrier. [6]

Vivek pathania at el. Measured Solvation Behaviour of Some Copper(I) Nitrate Complexes in Dimethylsulfoxide and Nitromethane at 298 k. IN WHICH Density

and ultrasound velocity were measured by DSA 5000M from Anton Parr at 298 K and 1 atmospheric pressure, Using ultrasonic velocity and density data, isentropic compressibility (κ_s) and apparent molal isentropic compressibility (κ_s, ϕ). Result shows that copper(I) electrolytes show less solvation in NM rich regions indicating structure breaking tendency of nitromethane. Extent of solvation in Cu(I) ions decreases in the order: [Cu(AN)4] + > [Cu(BN)4] + > [Cu(TU)4] + > [Cu(DMPhen)2] + > [Cu(Phen)2] + > [Cu(Bipy)2] [7]

Recent study done by BAL KRISHAN VERMANI on Solvation Behaviour of Some Copper(I) of perchlorate Complexes in Binary Mixtures of Dimethylsulfoxide with Dimethylacetamide and Dimethylformamide at 298 K in which he concludes limiting ion conductances (λ_{\pm}°) for various ions in the binary mixtures of DMSO with DMA and DMF have been calculated by an indirect method using [Bu₄N]BPh₄ as a reference electrolyte thus actual ionic radii (r_i) values for these copper(I) complex ions increase significantly with increase in DMA composition whereas the corresponding r_i values for these ions do not show a significant increase with increase in DMF composition that indicates no preferential solvation of copper(I) complex ions for DMF over DMSO, thus showing that DMA solvates the copper(I) complex ions more strongly than DMF. [8]

Esam A. Gomaa at el. In his study Ion association and solvation behavior of copper sulfate in binary aqueous–methanol mixtures at different temperatures (298.15, 303.15, 308.15 and 313.15) K shows that conclude that, the extent of ion-pairing in salt solutions under study depends upon the nature of the ion-solvent interaction taking place in the solution. Moreover it depends on the dielectric constant and the properties of the medium. The association increases as the temperature increases and as the proportion of organic solvent increases. [9]

A study done by G. Senanayake at el. On Competitive solvation and complexation of Cu(I), Cu(II), Pb(II), Zn(II), and Ag(I) in

aqueous ethanol, acetonitrile, and dimethylsulfoxide solutions containing chloride ion with applications to hydrometallurgy have been done in a range of aqueous ethanol (EtOH), acetonitrile (AN), and dimethylsulfoxide (DMSO) compositions containing excess chloride ion which is based on reduction potential and single ion activity of Cu(I), Cu(II), Pb(II), Zn(II), and Ag(I) concludes that decrease in metal ion activity in mixed aqueous solvents containing Cl⁻ is greater than that in concentrated aqueous chloride salt solutions, according to the strength of the chloro- or solvo-complex. These fundamental changes lead to applications in the extraction of metal ion complexes and promote the dissolution of AgCl, PbCl₂, and CuCl in aqueous DMSO containing Cl⁻. [10]

Dip Singh Gill proposed Both Na⁺ and Cu⁺ are highly solvated in AN + DMF mixtures over the entire composition range. Cu⁺ is relatively more strongly solvated than Na⁺ in AN + DMF mixtures. In his work Viscosity and molar conductance of Bu₄NBPh₄, Bu₄NCIO₄, [Cu(CH₃CN)₄]ClO₄, NaClO₄ and NaBPh₄ have been measured in the concentration ranges 0.02 – 0.5 mol dm⁻³ and 0.0005 – 0.0065 mol dm⁻³ at 298.15 K in AN + DMF mixtures containing 0, 10, 20, 40, 60, 75, 80, 90, and 100 mol % DMF.

The viscosity data have been analyzed by the extended form of the Jones-Dole equation. Solvated radii (r_i) for Na⁺, Cu⁺ and ClO₄⁻ have been estimated by using Gill's modification of Stokes' law. The variation of B_± and r_i as a function of mol % DMF shows that both Na⁺ and Cu⁺ are highly solvated in AN + DMF mixtures [11]

Vivek Pathania et al. In his study Physico-chemical and biological studies of some Copper(I)

complexes in binary mixtures of polar solvents at variable temperature (298 K–318 K) and at 1 atmospheric pressure.

In the present studies, polar non-aqueous solvents like Dimethylsulfoxide (DMSO), Pyridine (Py), and Nitromethane (NM) were

extensively used in evaluating the physico-chemical properties of prepared copper(I) nitrate complexes using the experimentally obtained viscosity, conductance, density, and ultrasonic velocity data.

Ionic B values were evaluated using Jones-Dole equation to determine the extent of molecular interactions, indicating that complex Cu⁺ ions were more easily solvated by Py in DMSO + Py mixtures. But, the result followed opposite trend in DMSO + NM region. The thermodynamic function values showed the values of ΔH* as positive for all electrolytes and ΔS* as negative [12].

Dip Singh Gill reported study about Transport Studies of Some 1:1 Copper(I) Perchlorate Complexes in Acetonitrile-Dimethylsulphoxide Mixtures deduce theoretically that by using combined approach of molar conductances and viscosity measurements. All copper(I) complexes are solvated and the extent of solvation is stronger in AN and AN rich region than in DMSO and DMSO rich regions.

The ClO₄⁻ ion is poorly solvated in AN+DMSO mixtures. The viscosity results are in good agreement with the results obtained from the conductance studies.

The conductance data have been analyzed by using the Shedlovsky method and the viscosity data by the Jones–Dole equation to evaluate viscosity B-coefficients of various salts. [13]

Esam A. Gomaa presented Copper sulfate volumetric properties in EtOH-H₂O solvents. The standard partial molar volumes of transfer for copper sulfate are determined. An Anton Par Model 55 densimeter is used to measure the densities at 298.15 K, 303.15 K, 308.15 K, and 313.15 K of copper sulfate solutions in H₂O and in EtOH-H₂O. Kraus–Bray and Shedlovsky models of conductivity were used to analyze obtained conductance data. Increased ion solvent and solvent–solvent interactions is suggested by increasing the amount of ethanol which indicated by limiting molar conductance values.

The negative $\Delta G^\circ A$ values indicate that the association processes in all studied systems are spontaneous processes. The negative estimation of $(\Delta H^\circ A)$ demonstrating that the association processes is exothermic in nature. [14].

R C Thakur in his studies Thermodynamic study of copper sulphate and zinc sulphate in water and binary aqueous mixtures of propylene glycol concludes that ion-solvent interactions dominate over the ion-ion interactions in water + propylene glycol mixture at 308.15 K.

Partial molar volumes of copper sulphate and zinc sulphate have been determined in water and binary aqueous mixtures of propylene glycol (2,4,6 and 8% by weight of propylene glycol) at 303.15 K with the help of density measurements. Effect of temperature on the partial molar volumes was also analysed for these salts in water and binary aqueous mixtures of propylene glycol. Results obtained have been analysed by Masson's equation. [15].

Peter Kroneck studied The behaviour of the ^{63}Cu nuclear magnetic resonance signals in tetrahedrally coordinated copper(I) complexes has been investigated as a function of temperature, solvent and anion. The ^{63}Cu NMR measurements were performed at approximately 23.86 MHz on a multinuclei Bruker pulse spectrometer SXP 4-100 in an externally stabilized magnetic field of 2.114. [16].

Dip Singh Gill at el. Studied Molar conductances of a large number of copper(I) and cobalt(III) complexes, behaving as strong 1:1 electrolytes, have been measured in acetonitrile (AN) and n-butyronitrile (n-BTN) at 298.15K. The actual ionic radii (r_i) for various ions in solution have been calculated using a modified form of Stokes' law. The ionic radii (r_i) for various complex ions have been compared with the ionic radii of two reference ions, Bu_4N^+ and Ph_4B^- , which are not solvated in dipolar aprotic solvents, to throw light on the solvation behaviour of these complex ions. [17].

Dip Singh Gill and Dilbag Rana reported Attempts have been made to prepare some

novel copper(I) nitrate, sulfate, and perchlorate complexes Molar conductances of these complexes have been measured in organic solvents like acetonitrile (AN), acetone (AC), methanol (MeOH), N,N-dimethylformamide (DMF), N,N-dimethylacetamide (DMA), and dimethylsulfoxide (DMSO) at 298 K.

The molar conductance data have been analyzed to obtain limiting molar conductances (Λ_0) and ion association constants (KA) of the electrolytes. The results showed that all these complexes are strong electrolytes in all organic solvents. The limiting ionic molar conductances (λ_{i0}) for various ions have been calculated using $\text{Bu}_4\text{N}^+\text{BPh}_4^-$ as reference electrolyte. The actual radii for copper(I) complex ions are very large and different in different solvents and indicate some solvation effects in each solvent system. [18].

Avnesh kumari analyzed Molar conductances of $\text{Bu}_4\text{N}^+\text{BPh}_4^-$, $\text{Bu}_4\text{N}^+\text{ClO}_4^-$, Bu_4NI , Bu_4NBr , $\text{Bu}_4\text{NCF}_3\text{COO}$, Pr_4NBr , Et_4NI , Et_4NBPh_4 , NaClO_4 , NaBPh_4 , $[\text{Cu}(\text{CH}_3\text{CN})_4]^+\text{ClO}_4^-$ and CF_3COOAg measured in the concentration range $(1-120) \times 10^{-4}$ mol dm^{-3} in acetonitrile (AN) and binary mixtures of AN with n-butyronitrile (n-BTN) and DMA.

Ion-association in case of silver (I) salts is the largest and relatively more in case of AN + DMA mixtures. Limiting ion conductances and hence actual solvated radii ($r(i)$) in all solvent systems have been evaluated using a modified form of Stoke's law. Cu^+ , Ag^+ and Na^+ are highly solvated, CF_3COO^- , ClO_4^- , Br^- and I^- are poorly/moderately solvated and tetraalkylammonium ions (R_4N^+) and Ph_4B^- are not solvated in AN + n-BTN and AN + DMA mixtures. [19].

Daniel Ricardo Delgado at el. Received The preferential solvation parameters of sulfadiazine, sulfamerazine and sulfamethazine in ethanol + water binary mixtures were derived from their thermodynamic properties by means of the inverse Kirkwood–Buff integrals (IKBI) method.

The more solvation by ethanol in mixtures of similar co-solvent compositions could be due mainly to polarity effects. Finally, the preference of these drugs for water in ethanol-rich mixtures could be explained in terms of the bigger acidic behavior of water interacting with hydrogen-acceptor groups in the sulfonamides. [20].

Ahmed Sadeq Abou Elyazed in his work using conductometric technique, the specific conductivities (Ks) of bulk and nano- CuSO₄ solutions were measured in the range of mixed solvent (MeOH-H₂O) at 298.15 K in the absence and in the presence of Spiramycin adipate (SA) antibiotic. the molar conductance (Λ_m), the limiting molar conductance (Λ_0), the association constant (KA) and Gibbs free energy change (ΔG_A) were calculated by using Fuoss- Shedlovsky and Fuoss-Kraus extrapolation methods. [21].

Y. Chu. At el. Reported The kinetic energy dependence of the collision-induced dissociation of Cu(acetone)_x, x = 1– 4 with Xe are examined in a guided ion beam mass spectrometer. The total binding energy of the Cu⁺ (acetone)₂ complex reported by Deng and Kebarle using measured values by Jones and Staley (once properly anchored) is in very good agreement with the value derived from our measurements. The trends in the measured Cu(acetone)_x binding energies are explained in terms of sp hybridization effects and ligand–ligand repulsion in the clusters. [22].

N. S. Rannulu at el. Analyzed sequential bond dissociation energies of Cu+(imidazole) _x, where x = 1-4, concludes The trends in the binding of these complex provide insight into the structural and functional roles that various His and other ligands play in the behavior of metallo-proteins and metalloenzymes, and suggest that at least two ligands are required for structural stabilization, while additional ligands play either structural or functional roles. [23].

Min zheng at el. In his study Solute-solvent and solvent-solvent interactions were

investigated from the available solubility data by using the linear solvation energy relationships concept, variation of hesperidin solubility depended upon hydrogen bond basicity for ethanol (1) + water (2), hydrogen bond basicity and dipolarity/polarizability for isopropanol (1) + water (2), hydrogen bond basicity, dipolarity/polarizability and cavity term for n-propanol (1) + water (2), and dipolarity/polarizability and cavity term of PG (1) + water (2).

In intermediate composition and cosolvent-rich composition, hesperidin was preferentially solvated by the cosolvent (ethanol, isopropanol or n-propanol). [24] rotational constants and energetics for the Cu I (imidazole) _x complexes and their dissociation products, density functional theory calculations at the B3LYP/6-31G* level are p

Declaration by Authors

Acknowledgement: None

Source of Funding: None

Conflict of Interest: The authors declare no conflict of interest.

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How to cite this article: Jassi, Veneeta Anand. Review of solvation studies of copper (I) complexes in different solvents and at different temperatures. *International Journal of Research and Review*. 2024; 11(2): 93-101. DOI: <https://doi.org/10.52403/ijrr.20240211>
