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Green Synthesis of Adipic Acid & Determination of Ultrasonic Parameters in the Presence of Green Solvent Methanol

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ABSTRACT: Adipic acid was synthesized by the oxidation of cyclohexene using 30% hydrogen peroxide in a microemulsion in the presence of sodium vanadate as catalyst. The proposed green process is environmentally friendly since catalyst and surfactant are recycled and pure adipic acid is produced in high yield (70% to 79%). Microemulsions are used as a "green solvent" and give a better contact between the phases. Sodium Dodecyl Sulphate was used as a surfactant for the generation of the microemulsion since it enables the use of harmful organic solvents and phase-transfer catalysts to be avoided. Optimised operating conditions (temperature, reaction time, separation process) have been defined and applied to evaluate the industrial practicability. The main interest of the present work is the easy recovery of pure adipic acid and the reuse of the reaction media (surfactant and catalyst). This shows promise for developing a future green industrial process that will enable greenhouse gas emissions (N2O), among others, to be reduced.

KEYWORDS: Ultrasonic parameters, Adipic acid, Methanol, Ultrasonic interferometer.

I. INTRODUCTION:

Green synthesis is an environmentally friendly method presenting a different way of thinking in chemistry intended to eliminate toxic waste, reduce energy consumption, and to use ecological solvents (water, ethanol, ethyl acetate, etc.) Green synthesis of metallic nanoparticles has become a new and promising field of research in recent years. Green synthesis of nanoparticles has gained significant importance in recent years as it has several merits such as it is simple, cost-effective, nanoparticles formed have good stability, less time consumption, non-toxic by-products, environment-friendly and can be easily scaled up for large-scale synthesis. Chemical synthesis methods lead to the presence of toxic chemical species adsorbed on the surface of nanoparticles therefore, green synthesis has attracted attention for the synthesis of various metal and metal oxide nanoparticles. Green synthesis approaches are found to be more reliable and economic route to synthesize these metal nanoparticles. This review aims to explain the advantages of various types of biomolecules as reliable, sustainable, and ecofriendly components for synthesizing metal and metal oxide nanoparticles.

[1] Adipic acid or hexanedioic acid is the organic compound with the formula $(CH_2)_4(COOH)_2$. From an industrial perspective, it is the most important dicarboxylic acid: about 2.5 billion kilograms of this white crystalline powder are produced annually, mainly as a precursor for the production of nylon. Adipic acid otherwise rarely occurs in nature, but it is known as manufactured E number food additive E355. Adipic acid is an alpha,omega-dicarboxylic acid that is the 1,4-dicarboxy derivative of butane. It has a role as a food acidity regulator and a human xenobiotic metabolite. It is an alpha,omega-dicarboxylic acid and a dicarboxylic fatty acid. It is a conjugate acid of an adipate

[2] Adipic acid is a white crystalline solid. It is insoluble in water. The primary hazard is the threat to the environment. Immediate steps should be taken to limit its spread to the environment. It is used to make plastics and foams and for other uses.

Adipic acid (AA) has immense practical use in industrial for the production of nylon-66, nylon-6, lubricant and plasticizer. In current industrial processes, AA is synthesized mainly by oxidation of KA oil using 50–60% nitric acid as oxidant and copper/ammonium metavanadate as the catalyst . However, this process emits nitrous oxide which can cause ozone depletion, acid rain, and global warming. Furthermore, the applicability of the phase-transfer catalyst in industrial scale is expensive. Obviously, we need to develop more sustainable AA manufacturing process which can avoid the use of toxic reagents and tedious products separation.

[3] Cyclohexane, cyclohexanol, cyclohexanone can be oxidized to produce AA without formation of any greenhouse gases. Oxygen, air, hydrogen peroxide (H_2O_2) are regarded as clean oxidant since they give water as the only byproduct. It is essential to use separable and reusable inexpensive catalysts for development of sustainable protocols. Various of solid supported catalysts, such as metal oxides, hollow structure silicates, carbon nanotubes (CNTs), and polyoxometalates (POMs), show remarkable performances in AA synthesis, due to the inherent adsorptive properties and tunable acidity.

Some alternative bio-derived AA processes have been extensively reported for synthesizing AA by oxidizing lignocellulosic biomass derived chemicals, e.g., hemicellulose, cellulose, and lignin. Different processes including glucose to glucaric acid process, hydroxymethylfurfural (HMF) to furan dicarboxylic acid (FDCA) process, γ -valerolactone process, lignin and lignin-derived oils process, were reported for AA synthesis from biomass feedstocks . In the glucose conversion route, glucaric acid was formed as intermediate by oxidizing the glucose and further undergo hydrogenolysis to form AA . This reaction can be achieved in the presence of Au, Pt, and Pd catalysts. In the FDCA process, FDCA were formed as intermediates by oxidizing the HMF and was further hydrogeneolyzed to form AA . Noble Pt and Au metals-based catalysts were reported most effective for this reaction.

[4] Surfactant, also called surface-active agent, substance such as a detergent that, when added to a liquid, reduces its surface tension, thereby increasing its spreading and wetting properties. In the dyeing of textiles, surfactants help the dye penetrate the fabric evenly. They are used to disperse aqueous suspensions of insoluble dyes and perfumes. The surface-active molecule must be partly hydrophilic (water-soluble) and partly lipophilic (soluble in lipids, or oils). It concentrates at the interfaces between bodies or droplets of water and those of oil, or lipids, to act as an emulsifying agent, or foaming agent. Other surfactants that are more lipophilic and less hydrophilic may be used as defoaming agents, or as demulsifiers. Certain surfactants are germicides, fungicides, and insecticides. Surfactants are used in corrosion inhibition, in ore flotation, to promote oil flow in porous rocks, and to produce aerosols.

[5] Sodium dodecyl sulfate (SDS) or sodium lauryl sulfate (SLS), sometimes written sodium laurilsulfate, is an organic compound with the formula $CH_3(CH_2)_{11}OSO_3Na$. It is an anionic surfactant used in many cleaning and hygiene products. This compound is the sodium salt of the 12-carbon an organosulfate. Its hydrocarbon tail combined with a polar "headgroup" give the compound amphiphilic properties and so make it useful as a detergent. SDS is also component of mixtures produced from inexpensive coconut and palm oils. SDS is a common component of many domestic cleaning, personal hygiene and cosmetic, pharmaceutical, and food products, as well as of industrial and commercial cleaning and product formulations. SDS is synthesized by treating lauryl alcohol with sulfur trioxide, oleum, or chlorosulfuric acid to produce hydrogen lauryl sulfate. Lauryl alcohol can be used in pure form or as a mixtures of fatty alcohols. When produced from these sources, "SDS" products are a mixture of various sodium alkyl sulfates with SDS being the main component. For instance, SDS is a component, along with other chain-length amphiphiles, when produced from coconut oil, and is known as sodium coco sulfate (SCS). SDS is available commercially in powder, pellet, and other forms (each differing in rates of dissolution), as well as in aqueous solutions of varying concentrations.

[6] It forms a dihydrate $Na_3VO_4 \cdot 2H_2O$. Sodium orthovanadate is a salt of the VO3−4 oxyanion. It is a colorless, water-soluble solid. Sodium orthovanadate is produced by dissolving vanadium(V) oxide in a solution of sodium hydroxide:

$$V_2O_5 + 6\ NaOH \rightarrow 2\ Na_3VO_4 + 3\ H_2O$$

The salt features tetrahedral VO3−4 anion centers linked to octahedral Na^+ cation sites

Like many oxometalates, orthovanadate is subject to a number of reactions, which have been analyzed by V NMR studies. At high pH, VO3−4 ions exist in equilibrium with HVO2−4. At lower pH's, condensation ensues to give various polyoxovanadates. Ultimately, decavanadate is formed Sodium Orthovanadate is an inhibitor of several phosphatases and kinases. General inhibitor of tyrosine phosphatases, a large family of enzymes that catalyze the removal of phosphate groups from tyrosine residues.

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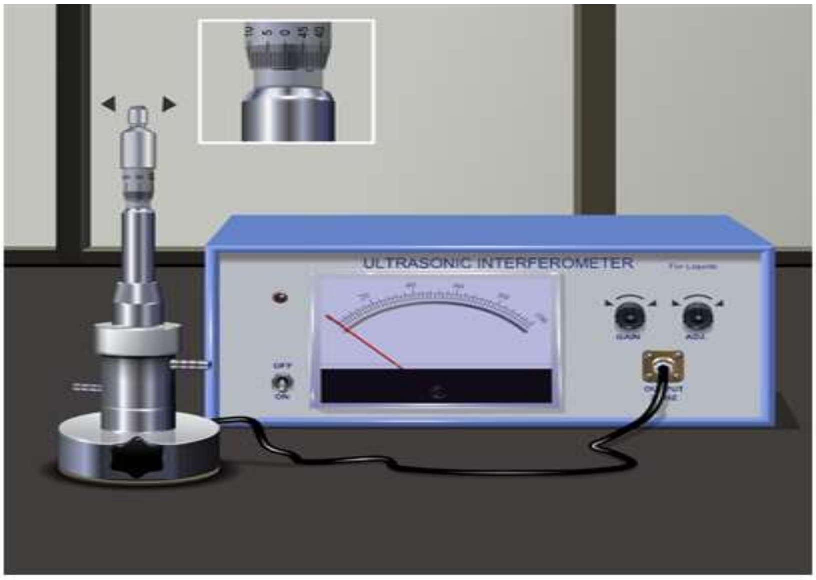
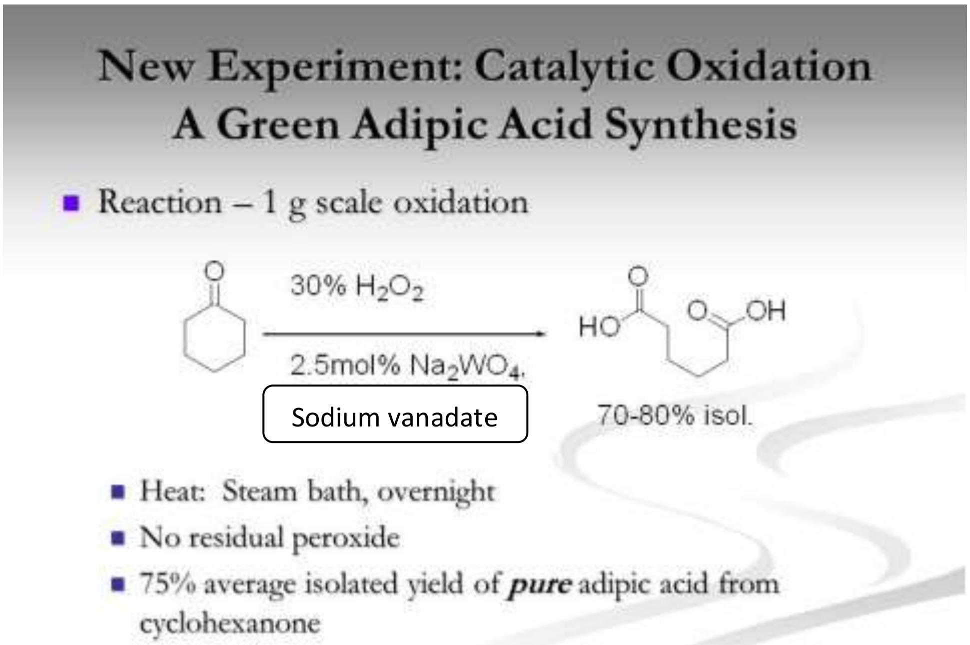
II. ULTRASONIC INTERFEROMETER:



III. EXPERIMENTATION:

Adipic Acid is Synthesized by the oxidation of cyclohexene using 30% Hydrogen Peroxide in Microemulsion in the presence of sodium vanadate as catalyst in the presence of Sodium dodecyl sulphate as Surfactant.



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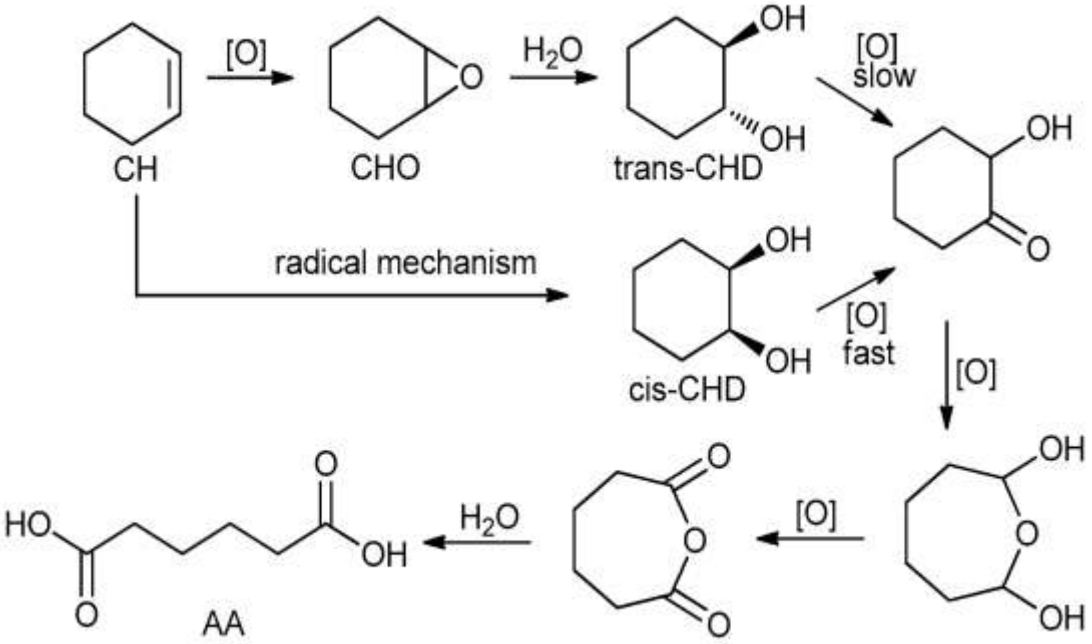

IV. OBESERVATIONS (TABLES & GRAPHS):

The values of sound velocity (U), along with the derived parameters, such as isentropic compressibility (K_S), molar compressibility (W), acoustic impedance (Z), molar sound velocity, relative association (R_A), intermolecular free length (L_f).

Table 4.2.1: Values of ultrasonic velocity, U (ms^{-1}), K_S (m^2N^{-1}), W ($N^{-1}m^{-1}$), Z ($Kgm^{-2}\ s^{-1}$), R ($m^{-8/3}\ s^{-1/3}$) and L_f (m) for, adpic acid in different weight percentage of methanol (green solvent) at 298.15 K.

Conc. Mol dm^{-3}	**U**	**K_s x 10^{10}**	**W**	**Z $X10^{-4}$**	**R**	**R_A**	**L_f $X10^{10}$**
1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Adipic acid in 5wt% methanol							
0.006	1522.8	4.24	0.40	154.84	0.22	1.2	5.23
0.008	1524.0	2.23	0.40	155.64	0.23	1.4	5.24
0.02	1528.8	4.22	0.40	155.73	0.23	1.4	5.21
0.04	1529.6	4.21	0.40	155.78	0.24	1.5	5.21
0.06	1530.0	4.20	0.40	155.80	0.24	1.5	5.20
0.08	1530.4	4.19	0.40	155.91	0.24	1.2	5.19
Adipic Acdi in 10wt% methanol							
0.006	1543.6	4.05	0.41	159.93	0.22	1.6	5.13
0.008	1544.4	4.04	0.41	160.07	0.22	1.7	5.13
0.02	1546.8	4.03	0.42	160.34	0.24	1.7	5.12
0.04	1547.2	4.02	0.42	160.47	0.25	1.7	5.12
0.06	1550.8	4.01	0.42	160.89	0.25	1.8	5.11
0.08	1556.8	3.97	0.42	161.62	0.26	1.0	5.10
Adipic Acid in 15Wt% methanol							
0.006	1572.0	3.83	0.43	166.20	0.22	1.8	5.02
0.008	1575.6	3.81	0.43	166.63	0.22	1.8	5.01
0.02	1578.0	3.80	0.44	166.76	0.25	1.9	5.01
0.04	1579.6	3.79	0.44	167.15	0.26	1.6	5.01

0.06	1580.8	3.78	0.44	167.29	0.26	1.6	4.9
0.08	1581.6	3.77	0.45	167.47	0.27	1.0	4.9

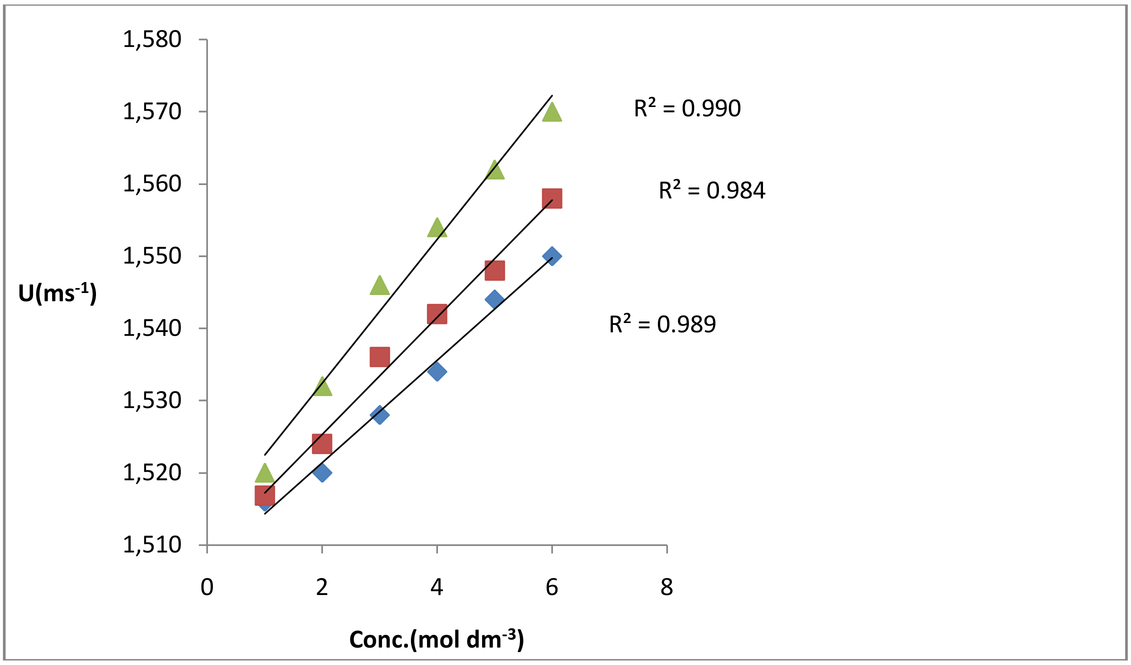

Fig-4.2.1(a) Plot of Ultrasonic velocity vs concentration In 5 wt% methanol solution.

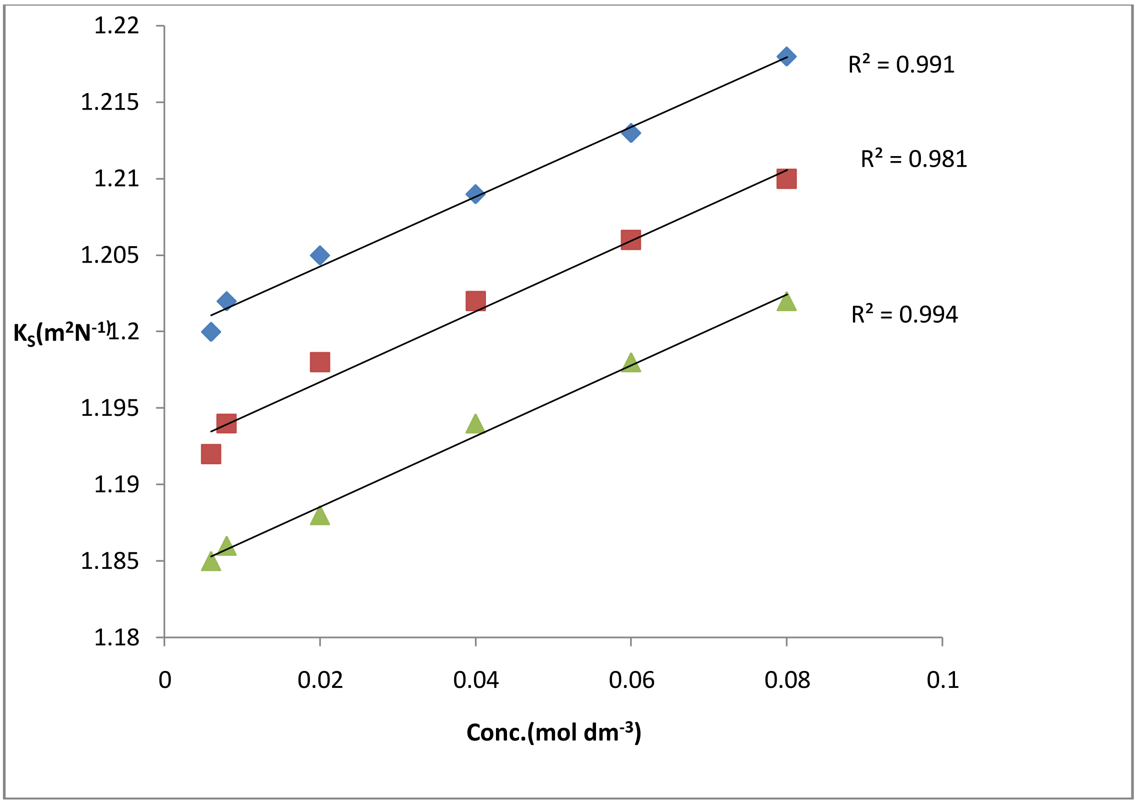

Fig-4.2.1(b) Plot of K_S vs concentration of adipic acid in different compostions of methanol solutions.

V. CONCLUSION:

- The Present work has been undertaken with a view to investigating the solvent effects of aqueous methanol on adipic acids in presence of surfactants from the, ultrasonic, measurements.
- The apparent and partial molar properties with other related properties of the solutions of the amino acids were evaluated from the density measurements in different compositions of aquo-organic binary solvents at different temperatures.
- The isentrepic compressibility, expansibility and various acoustic parameters of the solutions of adipic acids were derived from ultrasonic velocity measurements.
- The higher $V^0 Ø$ values for adipic acid in all solvents in presence of surfactants at all temperatures suggest that it is more solvated.
- The decrease in $V^0 Ø$with increase in temperature is attributed to increase in solvation of solutes.

SOME OF THE ADVANAGES FROM THE ABOVE RESULTS

a) The synthesis of Adipic acid by using greener chemicals is very much safe to the environment.
b) Ultrasonic velocity measurements give an idea about the sustainability of adipic acid.
c) The ultrasonic parameters give information about the pharmaceutical uses of adipic acid.
d) The adipic acid can be used in various synthesis which can be proved from the above observations.

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