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Replacement of classical experiments by experiments involving nanomaterials as greener alternatives

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ABSTRACT: Chemistry is indissolubly linked to our daily life. There are lot of classical experiments or conventional methods are available to synthesize chemical compounds or products. But the traditional or classical experiments has many disadvantages like air pollution, expensive, toxic to human health etc. Mainly, hazardous side products are responsible for the environmental pollution which is the most worrying issue now a days. To solve this problem scientists introduced greener methods which can avoid such difficulties. Replacement of classical experiments by experiments involving nanomaterials may be a such type of greener way. It was observed that many experimental methods involving nanoparticles are environment friendly as well as cost effective. This review discusses some key examples of nanomaterial-based experiments and emphasize on adopting such environment friendly practices in research for more environmentally conscious future.

I. INTRODUCTION

Classical qualitative analysis is performed by adding one or a series of chemical reagents to the analyte. For qualitative analysis the separated compounds were then treated with reagents that could be recognized by either colour, by their boiling points, their solubility in a series of solvents, their optical activities or their refractive indices. For quantitative analysis, the amount of analyte was determined by gravimetric or titrimetric measurements. By observing the chemical reactions and their products, one can deduce the identity of the analyte. The added reagents are chosen so that they selectively react with one or a single class of chemical compounds to form a distinctive reactions product. Normally the reaction product is precipitate or a gas, or it is coloured. Examples of some classical experiments are:
The determination of copper in brass, Brady's test for aldehydes and ketones, Measuring the amount of vitamin C in fruit drinks, Viscosity measurement, Making a mirror using silver nitrate and sugar, Titration-practical, Preparation of an organic liquid, Rates of reaction, Flame test for detection of cations and anions.

II. DISCUSSION

A. Advantages of classical chemical reactions:

Procedure is simple and accurate, The equipment needed is cheap, Methods are based on absolute measurements and Specialized training is not required.

Disadvantages of classical chemical experiments:

Hundreds of tons of hazardous waste are released to the air, water and land by industry every hour of every day, These is not cost effective, These methods may produce hazardous products and These are not environment friendly.

B. Green chemistry:

Green chemistry is defined as the "design of chemical products and processes to reduce or eliminate the use and generation of hazardous substances" [1, 2]. The concept of green chemistry were first developed at the beginning of the 1990s [3]. Paul Anastas is known as the "Father of green chemistry" for his work on green chemistry.
The concept of green chemistry is based on twelve principles. the twelve principle of green chemistry is introduced by Paul Anastas and John Warner in the year 1998 [4]. These principles are aim to guiding a framework for designing of new chemical products and processes, applying to all aspects of chemical classical reactions from raw material used to efficiency to reducing or removing hazardous material from the synthesis, the safety of transformation, the toxicity and the biodegradability of products [5, 6].

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Twelve principles of Green Chemistry:

Safer solvents and reaction condition, No derivatization, Use catalysts, Products biodegradabl, Strengthening analytical methodologies, Avoid chemical accidents, Prevention of waste, Maximum atom economy, Non-toxic products, Less hazardous synthesis, Minimum energy requirement and Use renewable feed stock.

Green chemistry approaches:

Green synthesis is far better than conventional chemical methods [7] due to the advantages it gives:
Easy synthesis, Cost effective, Eco-friendly, Biocompatible, High stability and Easy available.

C. Nanotechnology:

Nanotechnology offers a perfect way to reduce the effects of chemical and physical processes and use nanomaterials to prevent any toxicity, thus reducing the riskiness of nanotechnology and also the conventional experimental methods[8]. Nanomaterials, defined as materials having a single unit sized between 1 and 100 nanometre (nm). Nanotechnology, defined as the manipulation of matter between one dimension sized from 1 to 100 nanometre (nm) [9].

Eco- friendly synthesis of nanoparticles:

We can synthesis nanoparticles by chemically and biologically. There are many unfavourable effects associated to the chemical synthesis methods due to the presence of toxic chemical absorbed on the surface. Eco-friendly or green alternatives to chemical and physical methods are biological ways of nanoparticles synthesis. We can synthesis nanoparticle in biological methods by using microorganisms, enzymes, fungus and plants or plants extracts [10]. Diverse microorganisms, both procaryotes and eucaryotes are used for synthesis of metallic nanoparticles viz, silver, gold, platinum, zirconium, palladium, iron, cadmium etc. Different biological agents react differently with metal ions leading to the formation of nanoparticles. Many microorganisms produce inorganic materials either intra or extracellularly.

1. Synthesis of nanoparticle using bacteria- example, silver nanoparticles are synthesized using microorganism by bio-reduction process.
2. Synthesis of nanoparticles using actinomycetes- example, Novel alkalotolerant Actinomycetes, Rhodococcus sp. Was used to synthesize gold nanoparticles.
3. Synthesis of nanoparticles using yeast- all yeast genera can accumulate different heavy metals. they have the ability to accumulate significant amounts of highly toxic metals.
4. Synthesis of nanoparticles using fungi- intracellular synthesis of nanoparticles by fungi: this method involves transport of ions into microbial cells to form nanoparticles in the presence of enzymes. Extracellular synthesis of nanoparticles by fungi: fungi produce the nanoparticles extracellularly by their enormous secretory components, which are involved in the reduction and capping of nanoparticles[11].

Chemical methods to synthesis of various nanoparticles [12]**:**

1. Synthesis of calcium oxide nanoparticles- To a mixture of ethylene glycol (12ml) and $Ca(NO_3)_2.4H_2O$ (6g), 1g of NaOH was added and the solution were stirred vigorously at room temperature for 10 minutes ; the gel solution was kept for 5 hours at static state. Afterwards it was washed using water and dried under vacuum drying. Finally the prepared CaO was calcinated at 700 degree centigrade for 3 hours.
2. Synthesis of silver nanoparticles- Silver nanoparticles have synthesized using several green methods such as- Seed-mediated growth method in presence of ionic liquids, Reduction method such as hydrazine reduction method, sodium borohydride reduction method, Ag nanoparticles can be synthesized by a green photocatalytic method in which reaction is conducted in water medium, Using photochemical green synthetic method calcium alginate stabilized silver nanoparticles can be prepared.
3. Synthesis of gold nanoparticles- Gold nanoparticles also synthesized using several green methods- The Au nanoparticles is prepared by the addition of $HAuCl_4$ to green tea leaves at room temperature, By green photocatalytic method in which the synthesis is conducted in water medium, Calcium alginate stabilized gold nanoparticles are prepared using a photochemical green synthesis.
4. Synthesis of rhodium nanoparticles- Rh nanoparticles can be prepared using several green methods such as- Hydrogen reduction method in water as solvent, Ethanol reduction method in an ethanol water mixture.
5. Synthesis of platinum nanoparticles- PtNPs can be synthesized using several green synthetic methods such as- Hydrogen reduction method in water as solvent, Ethanol reduction method in an ethanol-water mixture, Monodisperse green Pt nanoparticles were synthesized by using glucose as reducing agent and starch as protecting agent.

Application of nanoparticles:

Application of nanoparticles in biologyand medicine[13]:

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Fluorescent biological labels, Drug and gene delivery, Bio detection of pathogens, Detection of proteins, Tissue engineering and MRI contrast enhancement

Application of nanoparticles in industry:

In petroleum industry [14], In textile industry[15]

Nanotechnology offers a perfect way to reduce the effects of chemical and physical processes and use nanomaterials to prevent any toxicity, thus reducing the riskiness of nanotechnology and also the conventional experimental methods [16].

D. Determination of Vit-C

Classical method of determining vitamin C:

Vitamin C is L-Ascorbic acid. Ascorbic acid is a potent reducing and antioxidant agent that functions in fighting bacteria infections, in detoxifying reactions, and in the formation of collagen in fibrous tissue, teeth, bones, connective tissue, skin, and capillaries [17, 18]. We intake vitamins in our body by food, especially by daily consumption of fresh fruite and vegetables. Fruits and vegetables containing high vitamin C are black blueberry, leaves of parsley, tomatoes dried in sun, broccoli, kiwi fruits, fresh cauliflower, berries, citrus fruits (lemons, oranges etc), green spinach etc. Deficiency in ascorbic acid or vitamin C causes a disease called scurvy. Recommended safety limit of vitamin C intake to human body is 60 mg/ kg body weight / day in adults, to prevent scurvy and minimum requirement is 10 mg / kg body weight / day. Now we can determine the quantity of pure ascorbic acid in tablets of pharmaceuticals from the following [19] study.

Experimental part:

Required chemical reagents and equipment-

1. Iodine (I_2) solution, 0.1 (N).
2. Sodium thiosulphate $Na_2S_2O_3$, 0.1 (N).
3. Standard solution of potassium dichromate $K_2Cr_2O_7$, 0.1 (N).
4. Freshly prepared starch solutions, 1% and 2%.
5. Sulfuric acid H_2SO_4, 40%.
6. Ascorbic acid tablets.
7. 300 ml volumetric flasks.
8. Graduated glass pipettes 1 ml, 5 ml and 10 ml.
9. 50 ml automated burette.

In this process a broad calculative method had been introduced to determine quantitatively the amount of Vit-C. actually it was here seen that lot of hazardous materials had been introduced to go through the procedure.

Quantitative analysis of vitamin C by silver nanoparticle[20]:

Silver nanoparticle can be synthesized by reducing the silver salt ($AgNO_3$) using sodium borohydride ($NaBH_4$), trisodium citrate, and also with ascorbic acid which is a mild reducing agent.

The reducing character of ascorbic acid was used to determined the amount of ascorbic acid in the vitamin C sample.

Synthesis of AgNPs by ascorbic acid-

Silver nanoparticles can be prepared by milder reducing agent like ascorbic acid. This is a slow growth process. The formation of AgNPs can be confirmed by taking absorbance of the resultant colloidal solution. So the absorbance was measured.

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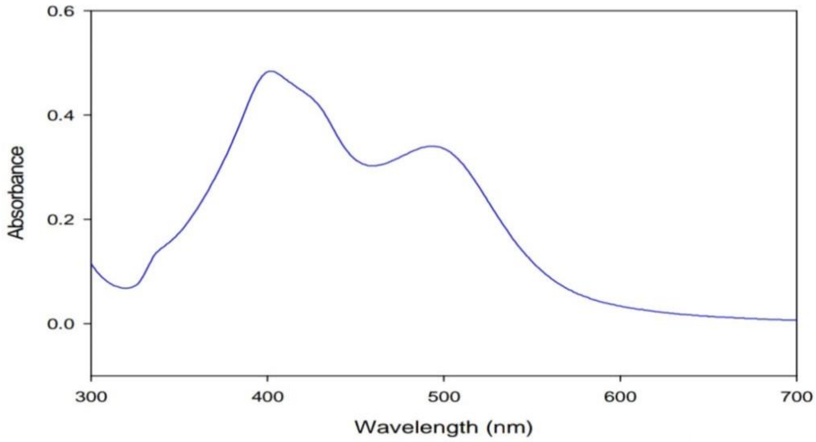

Fig. 2.1 the absorbance spectrum of AgNPs formed by ascorbic acid

The figure shows the absorption spectrum of silver nanocolloid prepared by ascorbic acid from $AgNO_3$.

Determination of ascorbic acid:

In a 25 ml volumetric flask 100 µl of silver seeds, 150 µl of TSC ($2.5* 10^{-2}$ M) and different conc. Of ascorbic acid were added. To this solution $AgNO_3$ (0.01 M) was added slowly 5 times (50 µl each time) with vigorous stirring. Then, a portion of that solution was transferred with in 2 min into a 1 cm spectrophotometric cell to record the absorbance.

Absorbance spectra for ceevit and nutrivit-C.

The absorbance spectra of Ag nanoparticles in the presence of the ascorbic acid in ceevit and nutrivit-C are shown in the figure below.

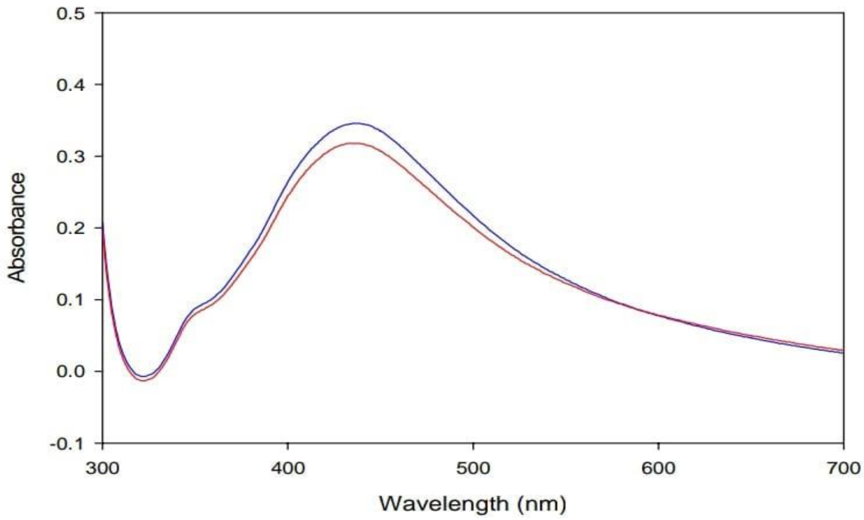

Fig. 2.2 Absorbance spectra of Ag nanoparticles in the presence of Ceevit (the blue line) and Nutrivit-C (the red line)

Fig 2, depicts that solution ceevit containing ascorbic acid shows absorbance at 0.336 at wavelength 435 nm and solution of nutrivit-C shows absorbance at 0.319 at wave length 432 nm.

Determination of ascorbic acid in vitamin C tablet in Ceevit and Nutrivit-C.

The absorbance value of silver nanoparticles in the presence of ceevit and Nutrivit-C is plotted on the calibration curve of Ag nanoparticles in the presence of different amount ascorbic acid containing solution to determine the concentration of the ascorbic acid present in the solution as shown in figure below.

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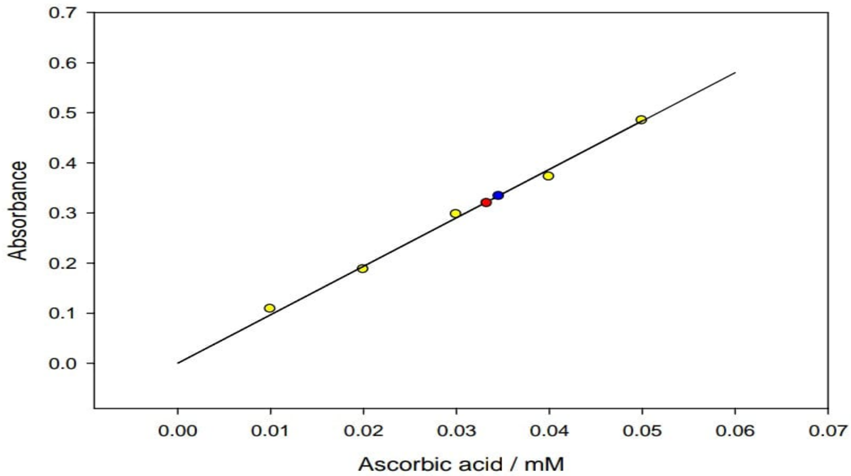

Fig. 2.3 Absorbance of Ag NPs in the presence of ascorbic acid vs concentration of ascorbic acid.

Figure 3 shows the calibration curve, from which the concentration of ascorbic acid present in Ceevit solution was found to be 0.0346 mM and for Nutrivit-C, the concentration was 0.0335 mM . The same procedure was followed for 2 times for both of the samples and the observed values are shown in the following table (Table 1.)

Table 1. Quantitative results of ascorbic acid in tablet samples

Samples	Ascorbic acid found (mg per tablet)	
	Claimed	proposed method
Ceevit	250	248.05
	250	253.91
	250	256.85
	250	242.90
Nutrivit- C	250	245.85
	250	248.05

In the study, silver nanoparticles and silver nanocolloid solution prepared chemically by the reduction of silver salt ($AgNO_3$). The nano species were characterized by UV-VIS spectrophotometry and SEM. The prepared silver nanomaterials were used to determine the amount of ascorbic acid present in real sample like vitamin C tablets (Ceevit and Nutrivit-C) that are available in the market of Bangladesh. The result obtained by the proposed method were in good agreement with that of claimed values of ascorbic acid by the pharmaceutical companies.
Both the results- from spectrphotometric determination using nps and conventional method gave the same result but the procedure involving nps showed less hazardous and less polluted in terms of using chemicals.

E. Preparation of Nylon
Classical method of nylon production:
The invention of nylon marked the beginning of the synthetic age as this fabric is the first one which is made entirely in laboratory. There are many commercial applications for nylon polymers which include fabric, fibres, shapes and films. Some properties of nylon are damage resistance, elasticity, strength, moisture resistance and quick drying.
Manufacturing process:
The reagent used in the production of nylon 6,6 is adipic acid (hexanedioic acid) and hexamethylene diamine [21]. The nylon 6,6 is produced by the slowly mixing of adipic acid and hexamethylene diamine.

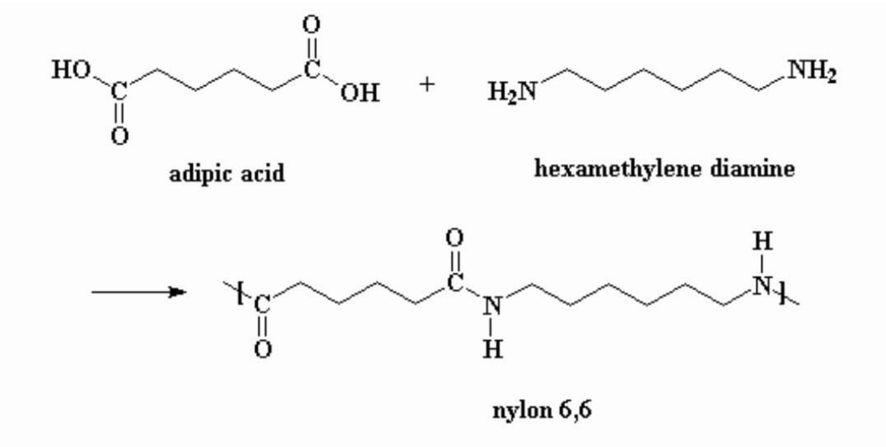

Fig. 2.4 synthesis of nylon 6,6.

Traditionally adipic acid is produced from the petroleum derived cyclohexane transformed to cyclohexanone and cyclohexanol mixture which is oxidised by nitric acid[22].
Unfortunately in this process of adipic acid production a large amount of N_2O releases, which causes global warming [23], furthermore the use of fossil sources and toxic or hazardous reagents is promoting environment pollution[24].
Nylon production using nanoparticles:
In recent years, several alternative synthesis of adipic acid were published, but a huge green step will be achieved using directly muconic acid (hexa-2,4-dienoic acid), from the biomass, as a starting compound.
The muconic acid can be mostly used as cis, cis- or trans, trans- isomer. The isomer cis, cis of muconic acid ((2Z,4Z)-Hexa-2,4-dienedioic acid) can be obtained from biomass, by the biotransformation of aromatics, de novo synthesis of glucose[25], or from lignin[26] with engineered pseudomonas putida, although the catalytic hydrogenation of trans, trans - muconic acid to adipic acid was more reported due to high stability of this isomer, which simplifies the kinetic study of the reaction excluding potential isomerization steps[27]. There is experimental evidence that the cis, cis- isomer isomerization to trans, trans and the successive hydrogenation to adipic acid.
The recently published hydrogenations of trans, trans- muconic acid, we can cite the reaction with-
Re/TiO2 in methanol at 210·C and 69 bar of hydrogen[28]
Pt/C in water at 7 bar of hydrogen and room temperature[29]
Pd/C at 50-70 C and 1 bar hydrogen[30].
[Because of the formation of intramolecular lactonized forms of cis, cis- muconic acid, it is not always quantitative]
The direct chemical transformation of cis, cis – muconic acid to adipic acid, by selective hydrogenation, recently, was achieved inseveral reaction conditions, using-
Pd/ C or Rh/ C as catalyst at 24C and 24 bar of hydrogen in ethanol solution[31].
In water with Ni/ alumina at 60C and 10 bar of hydrogen[32].
In methanol at 175C with Re/ C in absence of external hydrogen but in an inert atmosphere[33].
The scientific community is excited by the potential use of cis, cis – muconic acid as industrial platform, but, the reaction condition can be improved in terms of sustainability, reducing the temperature and / or hydration pressure, selecting an appropriate solvent and a metal catalyst greener, less expensive and not hazardous. So, to sum, combination a biotechnological step for the fermentative production of the bio- based cis, cis- muconic acid with its catalytic conversion to adipic acid could be a novel pathway to start greening the production of nylon.
Lignin is an underutilized waste- biomass, often used as a source of thermal energy, but essential in a sustainable industry transformation, its aromatic polymer nature makes it a resource for chemicals and building blocks, innovative products, and alternative fuels.
Several Pt and Pd nanoparticles are stabilized with lignin and used as catalysts at mild conditions in cross coupling, hydrogenation and reduction reactions.
In this way, we can suggest an important role for the lignin in a potential future production of nylon 6,6[34].

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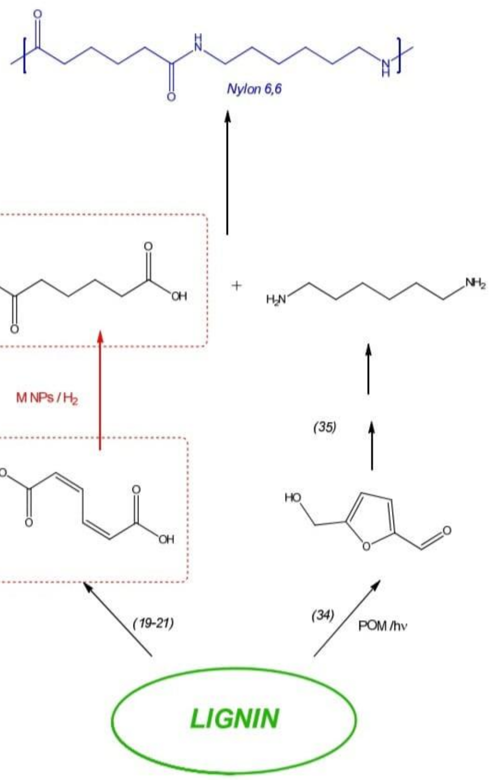

Fig. 2.5 pathway for green production of nylon 6,6 from lignin[34].

Various organic synthesis using nanoparticles as nanocatalysts:
Several reports shows that nanoparticles as catalysts perform amazingly in terms of reactivity, selectivity, and improving yields of products. Furthermore in comparison to their heterogeneous counter part nanoparticles can provide a large no of active site due their high surface-to-volume ratio[35].

Example of green nanocatalysts
Silver nanoparticles, Gold nanoparticles, Platinum nanoparticles, Rhodium nanoparticles, Iron nanoparticles, Calcium nanoparticles.

Example of organic synthesis-
Reduction reaction-

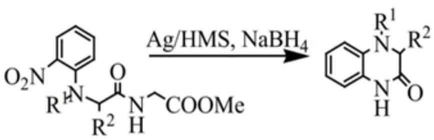

Fig. 2.6 Synthesis of amine-substituted MCR Scaffolds and Dihydroquinoxalinone Derivatives[35]

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Oxygen reduction by gold nanoparticles-

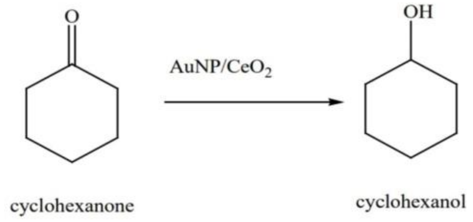

Fig. 2.7 synthesis of cyclohexanol from cyclohexanone[35].

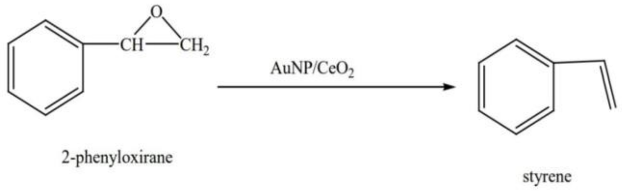

Fig. 2.8 synthesis of styrene from 2-phenyl oxirane[35]

Selective reduction of nitrobenzene-

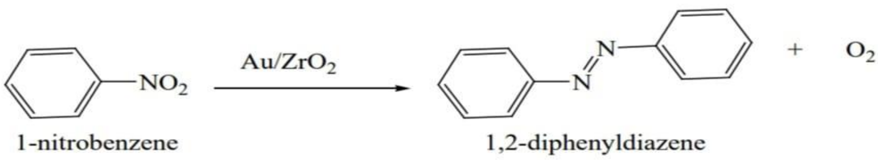

Fig. 2.9 synthesis of 1,2- diphenyldiazene from nitrobenzene[35]

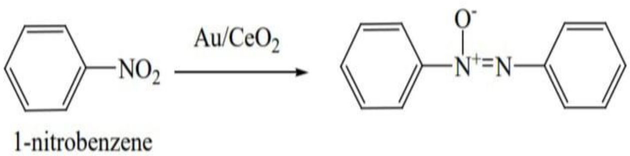

Fig. 2.10 Selective reduction of nitrobenzene[35].

Oxidation reaction-
Oxidation reaction by gold nanoparticles-

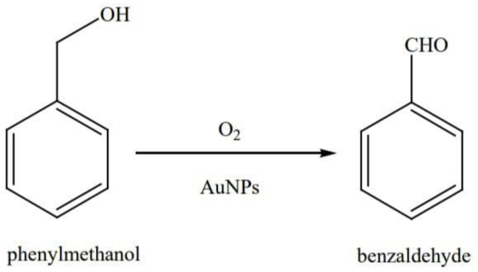

Fig. 2.11 synthesis of benzaldehyde by phenyl methanol[35].

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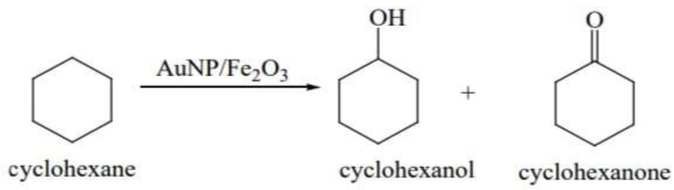

Fig. 2.12 synthesis of cyclohexanol and cyclohexanone from cyclohexane[35].

F. Oxygen production:

Gaseous oxygen is very important for the living organisms as it plays crucial role in metabolism of living organism and also has multiple application in agriculture, medical, and technological.

Different methods have been discovered for production of oxygen, including plants, oxygen concentrators and catalytic reactions[36].

Some classical method of oxygen production-

Oxygen production by photosynthesis-

Plant absorb sunlight, water and carbon dioxide and convert them into biomass and oxygen.

Reaction- **sunlight + n H_2O + n CO_2 = $(CH_2O)_n$ + n O_2**

Oxygen production by water splitting method-

In this method water splits and form O_2 and H_2 gas.

Reaction- **$H_2O = H_2 + O_2$**

Thermal decomposition of chlorates and perchlorates-

At very high temperature (above 400 degree centigrade), sodium chlorate (Na Cl O_3), and sodium perchlorate (Na Cl O_4) decomposes exothermally to produces gaseous oxygen and sodium chloride (NaCl)

Reaction- **2Na Cl O_3 = $3O_2$ + 2NaCl**

Oxygen generation by decomposition of metal peroxide-

Potassium superoxide (KO_2) reacts with water and produce hydroxide and O_2.

Reaction- **$4KO_2 + 2H_2O = 4KOH + 3O_2$**

Oxygen production by decomposition of H_2O_2-

Hydrogen peroxide is the best-known source of molecular oxygen. H_2O_2 decomposes exothermally and produce water and oxygen.

Reaction- **$2H_2O_2 = 2H_2O + O_2$**

Disadvantages of classical oxygen production-

There are many disadvantages of the classical synthesis method such as- relatively expensive, complexities in post-production processes, involve challenges, or generation of undesired products.

The disadvantage of using alkali peroxide is the risk of explosion as these reactions are highly exothermic.

Oxygen production using nanoparticles-

The decomposition of H_2O_2 over the surface of Pt nanoparticles occurs via a cyclic mechanism. Initially, one molecule of H_2O_2 reacts with Pt surface and form PtO. Another molecule of H_2O_2

Then reduces PtO to metallic Pt, releasing a molecule of H_2O and O_2. Particles that contain more PtO at their surface facilitate enhanced H_2O_2 decomposition and O_2 production(Naeem et al.; 2021).

III. CONCLUSION

In the above discussion we have focused on some green alternatives of classical experiments by the experiments involving the nanoparticles. It can be seen that we can synthesize nanoparticles by greener way using plant extracts, microorganism or using water as reaction medium. Further by the use of these nanoparticles we can do synthesis of products and can avoid undesired and hazardous side products and toxic chemicals which are not environment friendly and also toxic to living organism. That’s how we can introduce green chemistry in industry and also laboratory for more environmentally conscious future. Though all conversion from conventional methods to green methods have yet to be discovered

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