



**Miranda House
University of Delhi**

INSPIRE INTERNSHIP PROGRAMME 2024

**Innovation in Science Pursuits for Inspired Research
An Initiative of DST, Govt of India**

8-12 JULY 2024

**THE GREEN
PATHWAYS**

**Offered by:
Chemistry Department**



THE GREEN PATHWAYS: CHEMISTRY WORKSHOP

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WHAT IS GREEN CHEMISTRY?

Chemists have served the mankind from time gone by and will continue to work for the benefit of humanity. The unending efforts made by chemists have made available to us lifesaving drugs and products like plastics, petrol, diesel, LPG, synthetic fabrics, elastomers, cosmetics, coolants, agrochemicals, dyes, paints, adhesives, food preservatives, fire retardants and surfactants etc. One of the most important contributions has been in the field of medicinal chemistry - helping in diagnosis of and designing drugs for the treatment of diseases, which has not only provided relief from suffering but has also increased the average life span of humans. Chemists continue to strive to synthesize more potent and less toxic pharmaceuticals. Solutions have been found for the treatment of dreaded diseases like cancer, tuberculosis, hepatitis and diseases of cardiovascular system etc.

Due to the initial lack of awareness, some of these activities resulted in the pollution of air, water and soil, depletion of natural resources like minerals and fossil fuel etc. Now the scientific community has realized that in the process of production of useful materials, the protection of environment, non-renewable resources and health of living beings is crucial for sustainability. Hence, several efforts have been devoted to design eco-friendly techniques involving replacement of non-renewable energy sources by renewable sources, hazardous chemicals, reduction in generation of wastes and reaction times as well as scientific disposal of chemical waste. And a result of these efforts is the emergence of “**Green Chemistry**”. The term *Green Chemistry* was coined in 1991 by a chemist from United States- Paul Anastas, who is popularly known as the Father of Green Chemistry.

Green chemistry, also called sustainable chemistry, is a philosophy of chemical research and engineering that encourages the design of products and processes that minimize the use and generation of hazardous substances. Thus, Green chemistry protects the environment, not by cleaning up, but by inventing new chemical processes that are eco-friendly and energy efficient and follow the principles of Green Chemistry. Green Chemistry emphasizes renewable starting materials for a bio-based economy.

The 12 Principles of GREEN CHEMISTRY

Green chemistry is an approach to chemistry that aims to maximize efficiency and minimize hazardous effects on human health and the environment. While no reaction can be perfectly 'green', the overall negative impact of chemistry research and the chemical industry can be reduced by implementing the 12 Principles of Green Chemistry wherever possible.

1. WASTE PREVENTION



Prioritize the prevention of waste, rather than cleaning up and treating waste after it has been created. Plan ahead to minimize waste at every step.

7. USE OF RENEWABLE FEEDSTOCKS



Use chemicals which are made from renewable (i.e. plant-based) sources, rather than other, equivalent chemicals originating from petrochemical sources.

2. ATOM ECONOMY



Reduce waste at the molecular level by maximizing the number of atoms from all reagents that are incorporated into the final product. Use atom economy to evaluate reaction efficiency.

8. REDUCE DERIVATIVES



Minimize the use of temporary derivatives such as protecting groups. Avoid derivatives to reduce reaction steps, resources required, and waste created.

3. LESS HAZARDOUS CHEMICAL SYNTHESIS



Design chemical reactions and synthetic routes to be as safe as possible. Consider the hazards of all substances handled during the reaction, including waste.

9. CATALYSIS



Use catalytic instead of stoichiometric reagents in reactions. Choose catalysts to help increase selectivity, minimize waste, and reduce reaction times and energy demands.

4. DESIGNING SAFER CHEMICALS



Minimize toxicity directly by molecular design. Predict and evaluate aspects such as physical properties, toxicity, and environmental fate throughout the design process.

10. DESIGN FOR DEGRADATION



Design chemicals that degrade and can be discarded easily. Ensure that both chemicals and their degradation products are not toxic, bioaccumulative, or environmentally persistent.

5. SAFER SOLVENTS & AUXILIARIES



Choose the safest solvent available for any given step. Minimize the total amount of solvents and auxiliary substances used, as these make up a large percentage of the total waste created.

11. REAL-TIME POLLUTION PREVENTION



Monitor chemical reactions in real-time as they occur to prevent the formation and release of any potentially hazardous and polluting substances.

6. DESIGN FOR ENERGY EFFICIENCY



Choose the least energy-intensive chemical route. Avoid heating and cooling, as well as pressurized and vacuum conditions (i.e. ambient temperature & pressure are optimal).

12. SAFER CHEMISTRY FOR ACCIDENT PREVENTION



Choose and develop chemical procedures that are safer and inherently minimize the risk of accidents. Know the possible risks and assess them beforehand.



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GREEN CHEMISTRY IN INDUSTRY

A SUCCESS STORY OF GREEN CHEMISTRY:

GREEN SYNTHESIS OF IBUPROFEN

Pharmaceutical industries are a booming business. One of the major concerns of these industries is that huge volumes of by products are generated during the synthesis of most drugs. This increases the cost of the product and management of the waste is a challenge. Pharma industries are now making efforts to reduce the waste produced and are modifying the processes of drug manufacture. Here is an account of the Green syntheses of Ibuprofen- an Analgesic and an Anti-inflammatory drug. Analgesics (pain killers) are a very well-known group of drugs that include aspirin, acetaminophen, and ibuprofen (Motrin, Advil). In addition to their analgesic properties, ibuprofen and aspirin are also used as anti-inflammatory drugs

Problem: The traditional industrial synthesis of ibuprofen was developed and patented by Boots Company of England in 1960s (U.S. Patent 3,385,886). This synthesis is a six-step process and results in large quantities of unwanted waste (chemical byproducts) that must be disposed of or otherwise managed. Much of the waste that is generated is a result of low atom economy of the reactions i.e. many of the atoms of the reactants not being incorporated into the desired product (ibuprofen) but into byproducts.

Solution: The BHC Company has developed and implemented a new greener industrial synthesis of ibuprofen that is only three steps (U.S. patent 4,981,995 and 5,068,448 both issued in 1991). In this process, most of the atoms of the reactants are incorporated into the desired product. This results in only small amount of unwanted byproducts (very good atom economy/atom utilization) thus lessening the need for disposal and mediation of waste products.

The Boot's syntheses (known as the brown synthesis) and Green Synthesis of Ibuprofen are outlined below; the columns on the left side indicate the atoms in the byproducts.

CONVENTIONAL SYNTHESIS OF IBUPROFEN

4H
2C
as
CH₃COOH

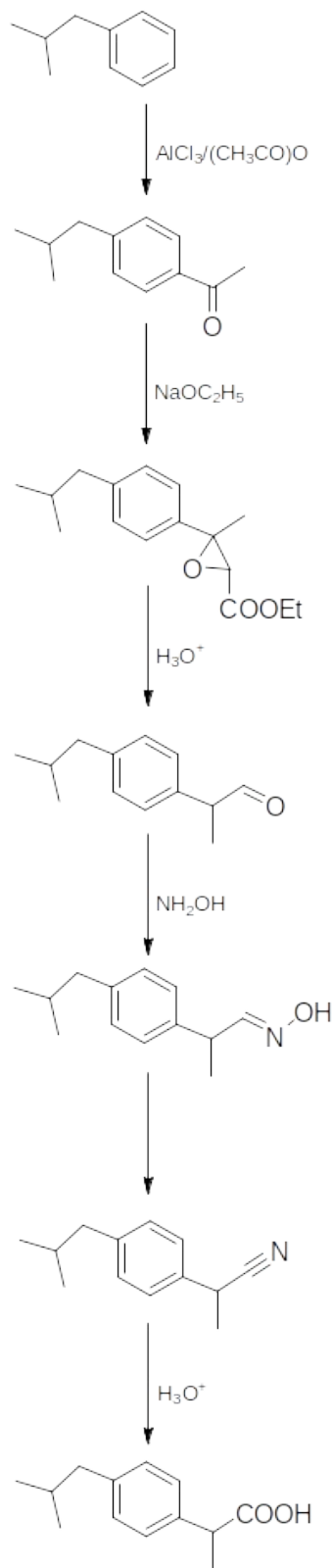
2C
6H
1O
1Cl
1Na as
EtOH
NaCl

3C
6H
3O
as
C₂H₅OCCOOH

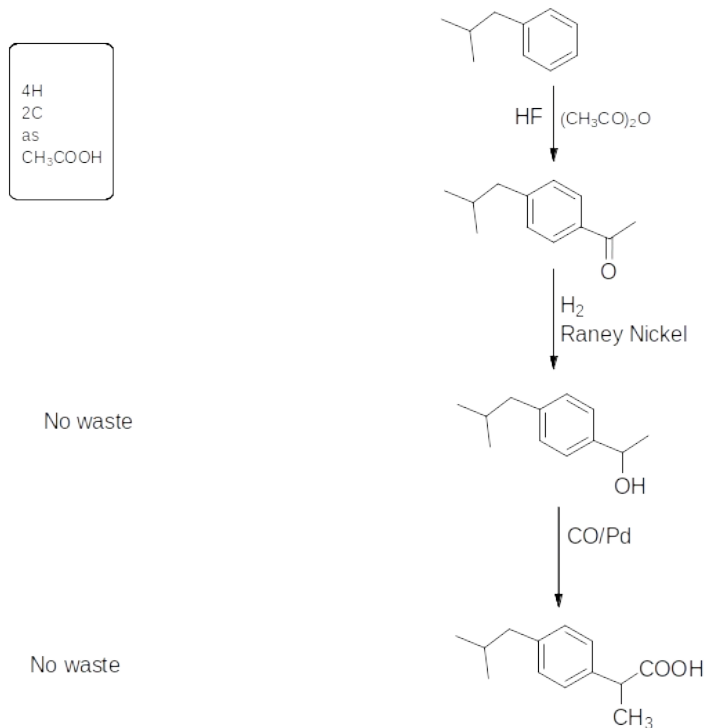
1O
2H
as
H₂O

1O
2H
as
H₂O

1N
3H
as
NH₃



GREEN SYNTHESIS OF IBUPROFEN



QUESTIONS

1. Find out the meaning of the following:
 - (i) Percentage yield of a reaction.
 - (ii) Atom economy of a reaction.
 - (iii) Can you give an example of a reaction with 100% atom economy?
 - (iv) Give any example of reaction with low atom economy.
 - (v) Renewable and non-renewable sources of energy.
2. Find out what CAS number of a chemical compound stands for.
3. After performing the experiments given below list the Green Chemistry principles that have been followed in each.
4. What do you think will be the benefits of adopting Green Chemistry in industry, R &D and academic institutes?
5. Can you give some suggestions for adopting Green Chemistry at academic institutes?

GREENER PROCEDURES FOR THE TEST OF N, S AND UN-SATURATION IN AN ORGANIC COMPOUND/ SPOT TESTS

Nature is the largest reservoir of organic compounds; most of these compounds are very useful for us, for example food (carbohydrates, proteins, fats, oils, and vitamins), clothing material (cotton, silk etc.), drugs (Penicillin and Quinine), dyes (Indigo) and rubber etc. Isolation and structure elucidation of these compounds is of utmost importance as many of these provide lead for the synthesis of related compounds which are useful for us. Students of chemistry are therefore given laboratory training to enable them to carry out scientific discoveries for benefit of mankind.

The first step towards structure elucidation of any compound is the detection of elements in it. Apart from C, H and O, many organic compounds also contain N and S, the latter two are known as extra elements (other types of elements Cl, Br, I, P etc. which may be present are also known as extra elements). Detection tests for un-saturation; functional groups etc are subsequently performed to arrive at the structure of a compound.

Spot Tests

(a)

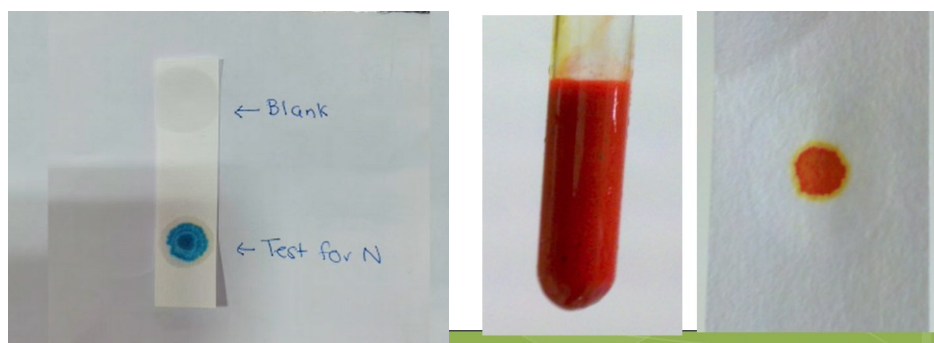
- ❑ **Spot tests** are based on chemical reactions that result in the production of any distinctive (generally colored) product, either as a spot on white paper, or in a droplet on a spot plate.
- ❑ **Spot tests belong to microanalytical chemistry.**

SPOT TESTS FOR DETECTION OF NITROGEN

Test for nitrogen

Procedure: Use Lassaigne's extract of the given compound for this test.

Conventional Test	Green/Spot Test
To the Lassaigne's extract (1-2 mL) in a test tube, add solid ferrous sulphate (50-70 mg). Boil gently for 30 seconds and acidify with dil. H_2SO_4 (3-5 mL). A Prussian Blue precipitate indicates the presence of N	Spot the following solutions (in given order) on a Whatmann filter paper: I. Freshly prepared conc solution of ferrous sulphate II. A spot of the Lassaigne's extract III. Dil. sulphuric acid Appearance of a blue colored spot shows the presence of N.



Spot test of N

Spot test: Azo Dye

TESTS FOR AMINES: AZO DYE TEST

Conventional Test	Green/Spot Test
To a cooled clear solution (obtained by reaction of acidic solution of compound with sodium nitrite), add slowly a cold solution of β -naphthol in dilute NaOH. Formation of an orange/red colored solid confirms the presence of an aromatic primary amine.	Spot the following solutions (in given order) on a Whatmann filter paper: I. Dilute solution of compound in water/alcohol II. Dilute hydrochloric acid III. Aq. Solution of sodium nitrite IV. Alkaline solution of β -naphthol Appearance of a orange-red spot shows presence of aromatic primary amines. Note: Blood red color fades, observe the color

	immediately.
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DRAWBACKS OF THE CONVENTIONAL METHODS FOR DETECTION OF EXTRA ELEMENTS

- Sodium is a very reactive and a hazardous metal
- Sodium cyanide is formed as the product of fusion of the nitrogen containing compound with sodium
- Enormous amount of chemical waste is produced

ROUGH ESTIMATE OF WASTE PRODUCED IN SCHOOLS OF DELHI

- Approximate number of students in 11th class: 300000
- Reagents used by each student for test for N and S: ~5ml
- Reagent used by students of Delhi: $300000 \times 5 = 1500000 = 1500$ liters

APPROXIMATE AMOUNT OF WASTE PRODUCED IN THE MODIFIED METHOD

- Each student uses ~ 0.2mL of reagent for tests for N and S
- Reagents used by 300000 students of Delhi: $300000 \times 1/5 = 60$ liters (25 times less!)

ADVANTAGES OF THE MODIFIED PROCEDURE

- Sodium has been replaced with Sodium acetate (a safer compound)
- The chemical tests are performed as spot tests, resulting in reduction in the consumption of chemicals and waste
- No heating is required – LPG saved

Similarly, the consumptions of chemicals and generation of chemical waste is drastically reduced in test for un-saturation and functional group tests by adopting spot tests rather than conventional test tube chemistry.

MICROWAVES AS AN ALTERNATIVE SOURCE OF ENERGY FOR CHEMICAL REACTIONS: SYNTHESIS OF ASPIRIN

Electromagnetic radiation is radiant energy that displays the properties of both particles and waves. The different types of electromagnetic radiation constitute the electromagnetic spectrum.

Frequency (ν) in Hz

γ -Rays	X- Rays	Ultra Violet Light	Visible Light	Infrared radiation	Micro waves	Radio waves
$>10^{19}$	10^{16} - 10^{17}	10^{14} - 10^{16}	10^{11} - 10^{14}	10^{11}	10^9	10^{-6} - 10^{-4}
	10^{-1}	1	10^0	10^5	10^8	

Wavelength (λ) in μm

A particle of electromagnetic radiation is called a photon. The relationship between the energy (E) of a photon and the frequency (ν) of the electromagnetic radiation is described by the following equation:

$$E = h\nu$$

Where h is the proportionality constant called Planck's constant. Frequency has units of hertz (Hz) or cycles per second (cps).

γ -Rays (gamma rays) are emitted from the nuclei of certain radioactive elements and because of their high energy, can severely damage biological organisms. X-Rays, somewhat lower in energy than γ -Rays are less harmful except in high doses. Low dose X-rays are used to examine the internal structure of organisms. The denser the tissue, the more it blocks X-Rays. Ultraviolet (UV) light is responsible for sunburns and repeated exposure can cause skin cancer by damaging DNA molecule in skin cells. Irradiation of a compound with UV radiation leads to excitation of electrons from a low energy level to a higher energy level. Visible light is the electromagnetic radiation we see. We feel Infrared radiation as heat. Exposure of a compound to IR leads to changes in vibrational and rotational energy levels in a molecule. We cook with microwaves and use them in radar. Now microwaves are used as a source of energy for chemical reactions. Radiowaves have the lowest energy (lowest frequency). We use them for radio and television communication. Radio waves are also used in NMR spectroscopy and in magnetic resonance imaging (MRI).

MICROWAVES

Microwave ovens work by using microwaves about 12cm in length to force polarised molecules in food to rotate. The interaction of these molecules undergoing forced rotation creates heat and the food is cooked. Polar molecules like water are electric dipoles and therefore rotate as they try to align themselves with the alternating electric field of the microwaves. Rotating molecules hit other molecules and put them into motion, thus dispersing energy. This energy when dispersed as molecular vibration in solids and liquids is heat.

Microwave energy, long used for heating food, is now finding new potentially useful applications in chemical technology. Microwave irradiation is very useful in rapid synthesis of a variety of organic compounds because of the selective absorption of microwave energy by polar molecules. Some materials for example glass, hydrocarbons and ceramics are nearly transparent to microwaves and therefore behave as good insulators in a microwave oven since they are heated to a limited extent. Metals reflect microwave energy, so metallic containers cannot be used in the microwave ovens.

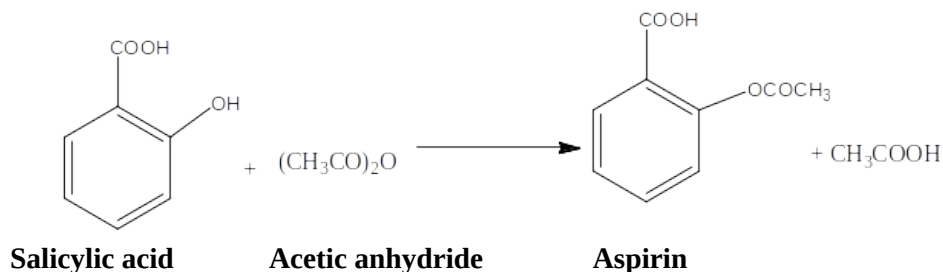
Microwave assisted reactions can be carried out under a variety of conditions: (i) Solution phase, (ii) on a solid support, (iii) without a solvent (solvent free). The main advantage of solvent free microwave reaction is the reduction or elimination of the use of volatile and toxic organic solvents. This facilitates easy isolation of products and promotes safety associated with handling the reaction in open vessels cumulating in chemical processes with unique attributes such as faster reaction kinetics, higher product yields and safer manipulations. The application of microwave enhancements in chemical synthesis has been extended to various disciplines of chemistry such as polymerizations, drug synthesis etc.

SYNTHESIS OF ASPIRIN

Chemicals Required:

Salicylic Acid 0.5g
 Acetic Anhydride 2mL
 Sulfuric Acid 1-2 drops

Chemical Reaction:



[A] Green Synthesis Using Microwave

1. In a clean dry 50 mL round bottomed flask, take 0.5g of Salicylic Acid.
2. Add to this 2mL of acetic anhydride, swirl and add cautiously 1-2 drops of sulfuric acid.
3. Place a small Teflon coated magnetic needle in the flask.
4. Fix an air condenser on the flask.
5. Heat the reaction mixture in the microwave at 70°C for about five minutes.
6. Let the reaction mixture cool down to room temperature.
7. Carefully take out the round bottomed flask and pour the reaction mixture into a 100mL beaker containing about 20-30 mL of ice cold water.
8. Stir with a glass rod. Filter the solid using vacuum pump.
9. Recrystallize, filter and determine the melting point of the dry compound.
10. The melting point of recrystallized sample of aspirin is 135°C.

[B] Conventional method:

When this reaction is carried out conventionally in the laboratory the reaction mixture needs to be heated for 30 mins on a water bath.

Lab test for checking the purity of aspirin

Test the alcoholic solution of recrystallized sample of aspirin with a solution of ferric chloride. If a purple color is obtained the sample is contaminated with salicylic acid.

SPOT TESTS FOR ANTIOXIDANT ACTIVITY OF SOME NATURAL PRODUCTS

FREE RADICAL OXIDATIONS WITH MOLECULAR OXYGEN: AUTO OXIDATION

When organic compounds are exposed to air, they react slowly with oxygen to give hydro peroxides. This slow oxidation reaction is called auto oxidation. Some compounds (e.g. unsaturated compounds, phenols, amines etc.) undergo faster oxidation in air as compared to others (e.g. Saturated and nitro substituted compounds etc.) This reaction is responsible for the slow deterioration in the air of food, rubber, polymers, gasoline and paints. The reaction is catalysed by light, which is why some compounds are stored in cans or dark coloured bottles. The ease with which a compound undergoes auto oxidation is directly related to the ease with which it forms free radicals. For that reason, auto oxidation takes place especially easily at allylic or benzylic positions.

Triacylglycerols also called triglycerides are compounds in which the three hydroxyl groups of glycerol are esterified with fatty acids. Triacylglycerides that are solids or semisolids at room temperature are called fats. Fats are usually obtained from animal sources and are composed largely of triacylglycerols with either saturated fatty acids or fatty acids with only one double bond. The saturated fatty acid tails pack closely together, giving the triacylglycerols relatively high melting points.

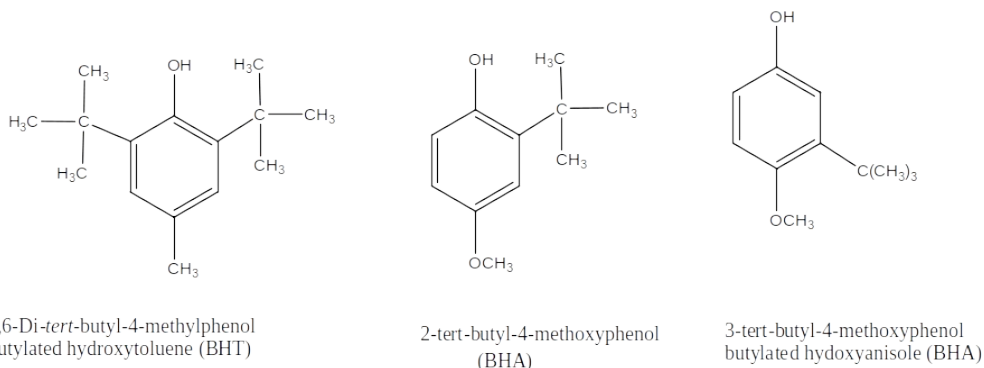
Liquid triacylglycerols are called oils. Oils typically come from plant products such as corn, soybeans, olives and peanuts. They are composed primarily of triacylglycerols with unsaturated fatty acids that cannot pack tightly together. Consequently they have relatively low melting points, causing them to be liquids at room temperature. Vegetable oils have become popular for food preparation because some studies have linked the consumption of saturated fats with heart diseases.

Polyunsaturated oils are easily oxidised by oxygen by means of a radical chain reaction. The reaction of fatty acids with oxygen causes them to become rancid. The unpleasant taste and smell associated with rancidity are the results of oxidation of alkyl hyper peroxides produced by the reaction of oxygen (a diradical) with the conjugated double bonds present in fatty acids to shorter chain carboxylic acids that have strong odors.

If these reactions are not stopped, the oils in food products stored at room temperatures in ware houses and on supermarket shelves are gradually oxidised and become rancid. The rancid oils taste bad and are also toxic. As the food processing industry became more and more centralised, it became necessary for foods to have a long shelf life. Therefore, the additives that breakup the

free radical chain (called **antioxidants**) are added to oils and a large number of other products to increase their life. The antioxidants could be natural or synthetic compounds

Antioxidants terminate these chain reactions by removing free radical intermediates and inhibit other oxidation reactions by being oxidised themselves. As a result, antioxidants are often reducing agents such as thiols, ascorbic acid or polyphenols are reactive towards radicals. Butylatedhydroxyanisole(BHA) and butylated hydroxyl toluene (BHT) give phenoxy radicals that terminate free radical chain reactions. As a result, they are used to retard free radical oxidation reactions in packaged food items like cakes, chips and kurkure etc. thereby increasing shelf life of the packaged food items. Similarly, vitamin C and E are used as natural antioxidants.



Antioxidants are widely used as ingredients in dietary supplements in the hope of maintaining health and preventing diseases such as cancer and coronary heart diseases. In addition to these uses of natural antioxidants in medicine these compounds have many industrial uses such as preservatives in food and cosmetics.

Antioxidants are also added to protect degradation of rubber, gasoline, adhesives, lubricants, plastics, elastomers and coatings from oxidation during high temperature processing and exposure to air.

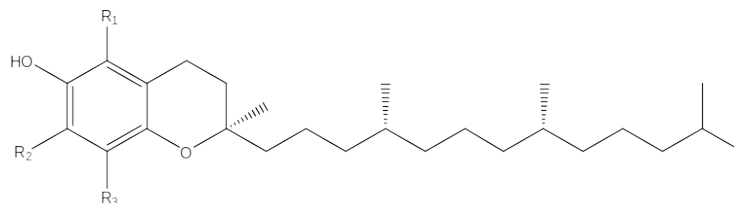
NATURAL ANTIOXIDANTS

Antioxidants have important role in the living beings. Unsaturated fats in the living beings are protected by vitamin E (α -tocopherol).It is the most important lipid soluble antioxidant and it protects membranes from oxidation by reacting with lipid radicals produced in the lipid per oxidation reaction. Tocopherols block the chemical damage due to radicals in the non polar environment.

Nuts like walnut, almonds, pistachio and oil seeds are rich in useful unsaturated oils. Vitamin E (an oil soluble compound) is present along with these in substantial amounts. Vitamin E is a powerful antioxidant and hence helps to increase the shelf life of these nuts. Vitamin-E also

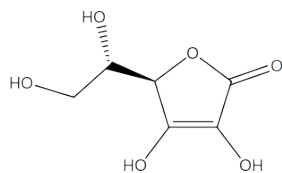
prevents biological membrane from reacting with oxygen. There are some people who believe that vitamin-E slows down the aging process.

Natural tocopherol exists as a mixture of four homologues: α , β , γ and δ available as a mixed product known as Mixed Tocopherol-which is also known as vitamin E.

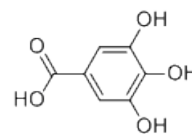


Homologue	R ₁	R ₂	R ₃
α -Tocopherol	CH ₃	CH ₃	CH ₃
β -Tocopherol	CH ₃	H	CH ₃
γ -Tocopherol	H	CH ₃	CH ₃
δ -Tocopherol	H	H	CH ₃

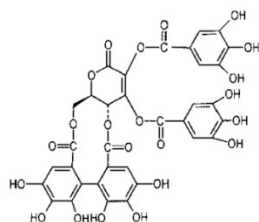
The damage due to radicals in the living beings, in polar or aqueous environments is controlled by vitamin C (L-ascorbic acid) and polyphenols etc. Ascorbic acid is a sugar acid synthesized in plants and in the liver of most vertebrates, but not humans. Human beings must constantly supplement their diet with vitamin C. The L-ascorbic acid plays an important role in preventing scurvy and in facilitating the healing of fractures etc. by its role in the synthesis of structural protein collagen. Amla is rich source of ascorbic acid (vitamin C), 445 mg/100g, and the overall antioxidant strength of amla may derive instead from its high density of ellagitannins such as emblicanin A (37%), emblicanin B (33%), punigluconin (12%) and pedunculagin (14%). It also contains, phyllanemblin other polyphenols: flavonoids, kaempferol, ellagic acid and gallic acid.



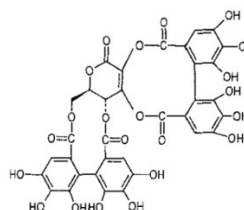
L-Ascorbic Acid



Gallic Acid



Emblicanin A



Emblicanin B

SPOT TESTS

To check the antioxidant activity of the extracts of given natural products by using potassium permanganate.

Natural Products Provided:

1. Amla juice
2. Walnut extract

Procedure:

Preparation of amla juice:

- Wash and cut amla (about 20g) in thin slices.
- Grind in the mortar and pestle to a fine paste.
- Filter out the juice.

Preparation of Walnut extract:

- Grind 10g of walnuts in a dry pestle and mortar.
- Add 5mL of petroleum ether to it and stir well with a glass rod.
- Decant out petroleum ether in a test tube.
- Remove the petroleum ether by heating in a hot water bath.

Important Precaution:

There should be no flame in the vicinity when petroleum ether is being evaporated.

Checking the antioxidant activity:

- On a Whatman filter paper strip put a spot of dilute potassium permanganate with the help of a thin capillary tube. Let the spot dry.
- Take a drop of amla juice from the sample bottle with the help of a thin capillary tube and place it over the spot of potassium permanganate.
- Observe the color change and also the rate at which the pink color of potassium permanganate disappears.
- Repeat the process with walnut extract and observe the color change.

LEARNING FROM NATURE

ISOLATION OF ACID BASE INDICATORS FROM NATURAL SOURCES

Natural Pigments

Pigments are responsible for both the green coloration of most leaves and the brilliant color changes seen in the foliage of some species of plants **as well as the colors of flowers and fruits.**

Pigments in the leaves and flowers:

Some of the common pigments are:

Chlorophyll a and b: green photosynthetic pigments found in the chloroplasts in leaves, immature branches, unripe fruits etc.

Carotenoids: they are mostly responsible for the yellows, oranges and orange-reds of some flowers and fruits.

Anthocyanins: these are the most common pigments of the flowers and are generally red, purple or blue in color. Levels of anthocyanins in vegetative leaves tend to vary with the highest levels seen in the spring and autumn. These are also present in many types of fruits. Anthocyanin color is influenced by acidity and production is stimulated by exposure to sunlight.

Betalains: these take the place of anthocyanins in many plants and are usually red to purple for e.g. the color of beetroots is due to betalains. Betalains and anthocyanins are never found together in the same species.

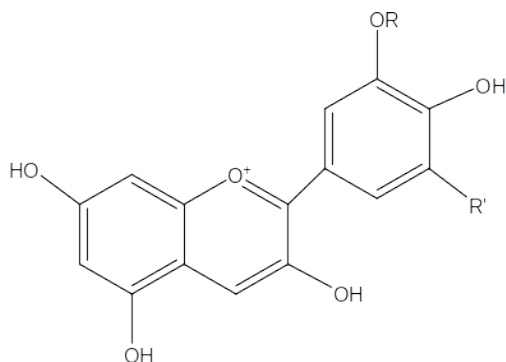
Pigments in fruits: many plants produce colorful and juicy fruits. Such fruits are typically green when unripe. As the fruit ripen, the chloroplasts in the skin turn to chromoplasts filled with bright yellow, orange and red carotenoids.

Deep red, pinkish red and purple fruits such as apples, grapes, black plum and berries usually have skins rich in anthocyanins. As chlorophyll levels drop in ripening fruits increased exposure to sunlight leads to the formation of these compounds. The blush seen on the sun exposed side of some apples, peach and mangoes is mostly due to the formation of anthocyanins.

NATURAL INDICATORS

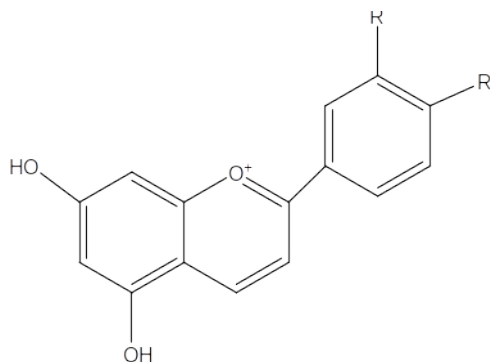
Many colored flowers and fruits contain anthocyanins and polyphenolics. These pigments show different color in acidic and alkaline medium and hence can be used as acid-base indicators.

Anthocyanins present in Black Plum (Jamun)



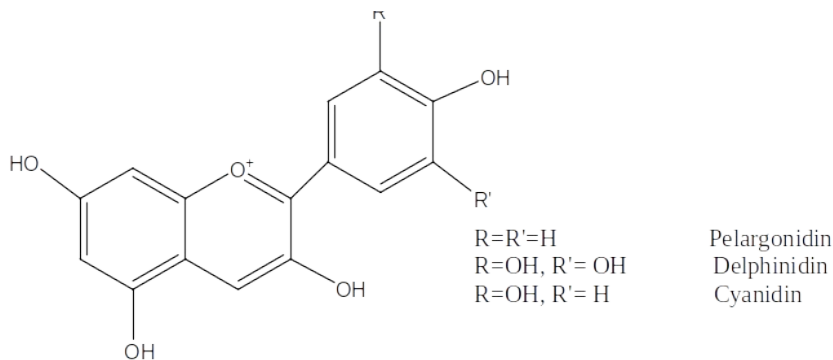
R=R=H	Cyanidin
,R=H, R'= OH	Delphinidin
R=CH ₃ , R'= OH	Petunidine
R=CH ₃ , R'= OCH ₃	Malvidin

Anthocyanins present in Rose



R=R'=OH	Cyanidin
R=H, R'= OH	Pelargonidin
R=OCH ₃ , R'= OH	Peonidine

Anthocyanins present in Pomegranate



(i) Isolation of indicator from rose petals and pomegranate

Procedure:

- Take fresh rose flower petals (20g).
- Grind the petals with 3-4 mL of water in a pestle and mortar.
- Filter and collect the aqueous extract.
- Similarly extract the red colour from pomegranate fruit

(ii) To make indicator paper using the above extract

- Take a small strip of Whatman filter paper No. 1.
- Dip it in the above extract.
- Take out the strip and dry in air.

(iii) Test using the above strip as acid base indicator

- Take the dry strip prepared above.
- On one corner of this strip spot dil. HCl using a capillary tube and note the color produced.
- On the other end of the strip spot a dil. Solution of NaOH and note the color change.

Note: This experiment can also be performed using the extract of black plum (jamun), pomegranate (anar), periwinkle (sada bahar flowers) and bogenvalliea as well.

SOLVENT FREE ORGANIC SYNTHESIS: SYNTHESIS OF AZOMETHINES

Traditional organic synthesis generally requires organic solvent as reaction media to facilitate the reaction as the organic reactants are usually soluble in these solvents. The disadvantage of using these solvents is that they are inflammable and many of them are toxic. Water an easily available, environmentally friendly solvent has limited application in organic synthesis because most organic reactants are water insoluble. Supercritical carbon dioxide is another “clean” solvent, but it usually requires high pressure reactor. Ionic liquids are now being used as green solvents. The best option is to carry out synthesis without using a solvent, i.e. solvent free reaction.

The goal in GREEN CHEMISTRY is to eliminate or minimize the use of volatile organic solvents in modern organic synthesis. Development of new synthetic methodology under solvent free conditions is an important area of research with growing popularity. Solvent free reactions reduce or eliminate solvent usage, simplify synthesis and separation procedures, prevent waste and avoid the hazards and toxicity associated with the use of solvents. (Benzene, carbon tetrachloride and chloroform are known to be carcinogenic, and methanol is highly toxic.)

SOLVENT FREE GREEN CONDENSATION REACTION BETWEEN *o*-VANILLIN AND *p*-TOLUIDINE (SYNTHESIS OF AZOMETHINE)

Chemicals required:

o-Vanillin 0.1g

p-Toluidine 0.06g

Chemical Reaction:



Azomethine

[A] Green Procedure:

1. Take 0.1g of *o*-vanillin on a clean dry watch glass
2. Add 0.06g of *p*-toluidine on the above solid in the watch glass.
3. Mix the two by continuously stirring the solid mixture using a dry glass rod.
4. An orange-colored liquid is obtained which solidifies.
5. Collect the solid.
6. Recrystallize, filter and determine the melting point of the dry sample.

[B] Conventional Method for Synthesis of Azomethines: These are prepared by heating equimolar quantities of aromatic aldehydes and amines at 160°C for about 30 minutes using anhydrous ZnCl₂ as a dehydrating agent.