INTRODUCTION
The struggle between prey and predators, hosts and pests is as old as life itself. The struggle between men and pests, which began long before the dawn of civilization, has continued without cessation up to date and will continue further, no doubt, as long as the human race endures. It is due to the fact that both men and certain pests constantly want the same thing at the same time. Pests are undoubtedly chief rivals of human beings, for the available food supply of the world.

Plants are economically important in many ways such as checking soil erosion, improving of soil fertility and providing food and shelter to many animals. Apart from these utilitarian aspects, plants are grown for beautification and aesthetic pleasures. In the Neolithic period sometime between 12000 and 10000 B.C. men took to growing of plants for their subsistence from hunting and food gathering habits.¹

Whenever plants are grown, whether for subsistence or meeting other necessities or even for aesthetic purpose, growers are keenly interested in having an assured yield or return. Accordingly, constant attention is paid to ensure proper growth and production, as successful cultivation of plants necessitates constant care and protection from dangers that may deter the cultivator from deriving full benefits from the economically important plants.

Pests have been known to cause damage to plants from ancient times. The pests are potential threats to the successful cultivation of crops and they can pose serious problems if adequate steps are not taken to protect the crops. Man suffers tremendous losses from the feeding and other activities of pests. Many pests feed on plants which man cultivates;
others feed on stored food grains, clothing or wood which are valuable for man. Late blight of potato caused by *Phytophora infestans* in Ireland in the 1840's resulted in a potato famine and the consequent migration of half a million people from Ireland to the U.S.A. One of the major causes of the Bengal famine of 1943, was the failure of the rice crop succumbing to the brown spot disease incited by *Helminthosporium oryzae*. Many obscure pest organisms at one time restricted in their area to wild plant hosts, have now become adapted to economically important plants wherever they are cultivated.

In recent years, it is an admitted fact that with the intensification in agriculture, a grower has to keep constant vigil against attack of pests to protect the crops and have good harvest. If measures for controlling pests, diseases, rodents, weeds etc. are not taken for the crops that are grown, they will not reach the desired level of production and as a consequence famine would engulf a huge chunk of population. Herein, comes the question of protection from the pests. Many pests are obnoxious or destructive. They attack various growing plants of value to men and feed on them, injure or kill them or introduce disease into them.

**THE PEST**

Insects and disease producing organisms are normally recognized as pests. Pest has been defined as; any organism detrimental to man or his property in causing damage significant of economic importance. This organism may be insect, arachnid, nematode, disease producing pathogen including fungi, bacteria, viruses, mycoplasma, weeds, angiospermic parasites, rodents, birds and other animals. In the animal kingdom animals ranging from an elephant or a monkey to nematodes may be pests.
Pests may also be defined as organisms, which consume, damage or destroy agricultural products either in the field or during storage. The term ‘pest’ is also extended to include weeds, which by competing with crop plants reduce yields.

Nature tends to scatter the plant species making it difficult for pests to spread and survive, whereas agriculture tends to concentrate millions of plants of a particular species in a limited defined area year after years.

Due to mutation in nature or due to the development of resistance against pesticides, often-new races or strains of pests appear which are virulent and destructive and cause extensive damage.

**DAMAGES CAUSED BY PESTS**

Damages caused by pests may be qualitative or quantitative or both. Quantitative loss is recorded when there is an overall reduction in yield or out turn. In case of qualitative damages, the gross yield may not be affected but the presence of markings, blemishes, wart or offensive odour may fetch much less price and the net income from unit area is reduced.

Damages caused to the plants due to attack of pests may again be either direct or indirect depending on the parts affected and the resultant effect. The pests is said to be direct when the host is damaged by its direct action in a direct or indirect manner. If infection does not cause any damage to the host but is affected in some other way, pest may be termed as indirect. Pests, however convey the idea or concept that they are controlled by suitable methods.

Insect pests have been a major constraint in agriculture production. Inspite of all advances made in crop protection technology, losses caused
by insect pests have increased with modernization of agriculture. It is estimated that globally 15.6% of attainable production is lost to pests (mostly insects) and the current losses due to pests in field crops have been estimated to be Rs.68,940 crores.²

CONTROL OF PESTS

Historically, pest control is very ancient. Pest control may be defined as any method or procedure employed to reduce the pest population and prevent damages caused by them. The control of pests signifies any action taken by men to mitigate or prevent losses caused by them. Control measures adopted for pest control may not always aim at the destruction of pests but also amelioration of damages caused by them.

There is increasing emphasis on integrated biological and chemical pest control methods to reduce the use of chemicals, which will mean less dangers of environmental pollution and opportunity for the emergence of resistant strains of pests.

There have been attempts to classify different control measures that are employed for protection of crops against pests. But a rigid clarification is difficult in view of the interdependence of methods upon one another. Broadly, there may be two approaches -

(1) Those methods, which are direct against the parasite to reduce population to a very low-level by chemical, biological, autocidal and cultural methods.

(2) Those methods in which the host is growing so that host plant is in a position to counteract attack of different parasites in an effective manner.
Methods of pest control may be presented as follows:-

(a) Protection of the host.
(b) Destruction of the pest.

The destruction of the pest includes chemical pesticides.

PESTICIDES

Pesticides are chemical substances used to kill organisms (bacteria, fungi, insects, nematodes, rats, birds etc.) which consume, damage or destroy agricultural products either in the field or during storage.

Pesticides are defined under the Federal Environmental Pesticides Control Act as including:

1. "Any substance or mixture of substances intended for preventing, destroying, repelling or mitigating any pest".

2. "Any substance or mixture of substances intended for use as a plant regulator, defoliant or desiccant."

Pesticides or agrochemicals are chemicals designed to combat the attacks of various pests on agricultural or horticultural crops.

Prior to World War II most of the pesticides were inorganic chemicals that were simple in nature and only a few insecticides were of plant origin. The discovery of DDT in 1939 revolutionised the concept of chemical methods of pest control. This was soon followed by the discovery of gamma isomer of BHC. It may be stated that the tremendous development in range of chemicals to be used as pesticide has made a definite impact on pest control. At present pesticides are frequently used for control of insects, arachnids, molluscs, nematodes, rodents, fungi, bacteria etc. The use of pesticides constitutes probably the most important method in spite of their limitations and adverse effect on the environment.
HISTORICAL ASPECTS OF PESTICIDES

Ever since the dawn of civilization man has continually endeavoured to improve his living conditions. In his efforts to produce adequate supplies of food man has been opposed by the ravages wrought by insect pests and crop diseases. The blasting mentioned by the prophet Amos (760 B.C.) was the same cereal rust disease, which is still responsible for enormous losses. The father of Botany, Theophrastus (300B.C.), described many plant diseases known today such as scorch, rot, scab and rust. There are also several references in the Old Testament to the plagues of Egypt for which the locust was chiefly responsible and even today locusts cause vast food losses in the near East and Africa.\(^6\) The major pests inhibiting the growth of agricultural crops are insects, fungi and weeds, and the idea of combating these pests by the use of chemicals is not new.

By the year 1850 two important natural insecticides were introduced; rotenone from roots of the Derris plant and pyrethrum from the flowers of a species of plant Chrysanthemum. These are still widely used insecticides. Many of the well-known poisons have been applied at one time or another for the control of insects and other pests. They were sometimes quite successful, although the hazards to the operators were great. Cyanide, generally as Hydrogen cyanide gas, has been used as a fumigant in buildings to kill bedbugs and wood-boring beetles. Initially this treatment proved a considerable success but after a time failures became apparent. These resulted from the development of resistant strains of the insect- this was the first reported example of resistance to an insecticide. In 1897 formaldehyde was introduced as a fumigant and in 1913 organomercurials were first used as fungicidal seed dressing against cereal smut and bunt diseases.\(^6\)
The 1930's really represent the beginning of the modern era of synthetic organic pesticides—important examples included the introduction of alkyl thiocyanate insecticides (1930), the first organic fungicide; salicylanilide (1931), dithiocarbamate fungicides (1934), two other protectant fungicides—chloranil (1938) and dinocap (1946). Other organic compounds used during this period were azobenzene, ethylene dibromide, ethylene oxide, methyl bromide, carbon disulphide as fumigants and phenothiazine, p-dichlorobenzene, naphthalene and thiophenylamine as insecticides. In 1939 Dr. Paul Muller discovered the powerful insecticidal properties of dichlorodiphenyl trichloroethane or DDT and following successful initial field tests in Switzerland against potato beetle. It was manufactured in 1943 and soon became the most widely used single insecticide in the world. Present fears about the long term deleterious effects of DDT and other organochlorine insecticides on ecosphere must not allow us to forget our tremendous debt to DDT. This compound controls louse-bourne typhus and is equally effective against malaria carrying mosquitoes. A closely related group of insecticides are the carbamate esters, first discovered by the Geigy Company in Switzerland in 1947, although the most generally effective member of the group carbaryl or sevin was not introduced until nearly a decade later. This is becoming of increasing importance as a possible replacement for DDT.

Properly constructed stores can keep out rats, and fungi and mites can be inhibited by controlling grain moisture content at < 13%. Insects are more difficult to control by physical methods and there is a need for the further development of safe insecticide for use on stored crops.
CLASSIFICATION OF PESTICIDES

Pesticides fall into three major classes: insecticides, fungicides and herbicides. There are also rodenticides (for control of vertebrate pests), nematicides (to kill microscopic eelworms), molluscicides (to kill slugs and snails) and acaricides (to kill mites).

Pesticides may also be divided into two main types, namely contact or non-systemic pesticides and systemic pesticides. Contact or surface pesticides do not appreciably penetrate plant tissues and are consequently not transported or translocated, within the plant vascular system. Their disadvantages are that they are susceptible to the effects of weathering (wind, rain and sunlight) over long periods and new plant growth will be left unprotected and hence open to attack by pests. In contrast, the systemic pesticides can effectively penetrate the plant cuticle and more through the plant vascular system.

TYPES OF PESTICIDES

PESTICIDES

ORGANIC

NATURAL

Example-
(1) Pyrethrum
    (Chrysanthemum flowers)
(2) Nicotine
    (Tobacco leaves)
(3) Rotenoids
    (Derris roots)

INORGANIC

SYNTHETIC

Example -
(1) DDT
(2) Aldrin
(3) Gammexane
(4) Baygon etc.
The pesticides can be grouped into following branches:-

1. Insecticides
2. Herbicides
3. Rodenticides
4. Fungicides
5. Nematicides
6. Bactericides
7. Defobiants

**MODE OF ACTION OF PESTICIDES**

The nervous system is characteristic of vertebrates and insect pests and carries signals from the various receptor sites (e.g. eyes, ears and nose) to the brain. The external environment also transmits information to the nervous system by means of discrete electrical impulses along a long fibre of neurons (or nerve cells) known as the axon, eventually reaching the brain, so that the appropriate response can be made to the received stimuli. The brain and spinal cord together are termed the central nervous system (CNS) and this sends out signals and communicates with the rest of the body by the peripheral nervous system.¹⁰⁻¹¹

The activity of the nervous system depends on the neurons capacity to maintain an unequal distribution of sodium and potassium ions on each side of the cell membrane. Under resting conditions, the electrical potential inside the membrane is negative with respect to the outside, and the concentration of Na⁺ ions inside the nerve cell is low, relative to the outside, whereas for the K⁺ ions the reverse is true. This situation arises from the fact that Na⁺ ions are actively transported out of the cell, while K⁺ ions more into it. This unequal distribution of ions on the two sites of the cell membrane gives rise to an electrical potential.
The transmission of the nervous impulse is therefore an electrical process in which the current is carried by ions. When the axon meets another neuron there is a junction called a synapse. Nerve impulses are transmitted at the synapse by the release of a chemical transmitter, acetylcholine.

When a nerve impulse arrives at the presynaptic membrane, acetylcholine is simultaneously released from the presynaptic cells and the chemical transmitter diffuses across the synaptic cleft to the postsynaptic membrane, where it binds to the acetylcholine receptor sites. The liberated acetylcholine must not persist in the synapse too long, otherwise there would be a continuous chain of nerve impulses. The transmitter is generally eliminated by combination with the enzyme acetylcholinesterase present in the postsynaptic membrane. [Fig. 1.1]

**Fig. 1.1: Schematic diagram of nerve synapse**

Acetylcholinesterase catalyses the hydrolysis of acetylcholine to choline which does not act as a transmitter of nerve impulses.

\[
(CH_3)_3\text{NCHCH}_2\text{OCOCOCH}_3 \overset{\text{Acetylcholinesterase}}{\longrightarrow} (CH_3)_3\text{NCHCH}_2\text{OH} + CH_3\text{COOH}
\]

Acetylcholine  
Choline
The combination of acetylcholine with the receptor causes the postsynaptic cell to pass an impulse; subsequently acetylcholinesterase hydrolyses the acetylcholine so that stimulation of the receptor ceases and the synapse in then available for release of a new transmitter.

Pesticides poison the enzyme so that it cannot catalyse the hydrolysis of acetylcholine to choline; consequently there is an accumulation of acetylcholine at the synapse, which permits the continuous transmission of nerve impulses, and effective nervous coordination breaks down. Thus, the insect pest suffers convulsions and paralysis and finally death.

SYNTHETIC PESTICIDES

Pesticides have proved important chemicals for agricultural production. It is estimated that insects, weeds, plant diseases, rodents and nematodes account for losses upto Rs. 20 crores annually in India alone.\textsuperscript{13} The use of pesticidal chemicals in agriculture makes it possible to save approximately more than one third of our crops. Pesticides by destroying pests, in fact help us in improving our economic conditions. Although there are some bad points, but production of agricultural products has increased tremendously with the use of pesticides all over the world. These synthetic pesticides have increased human diseases and polluted the environment. The increasing use of pesticides in the under developed countries is absolutely vital, if such countries are to obtain the greater supplies of food necessary to feed their large population adequately.

Some examples of synthetic pesticides are as follows-
1. Baygon

2. Dinoseb

3. Chloroneb

4. Imidon

5. Erbon

EFFECT OF PESTICIDAL RESIDUES ON ENVIRONMENT

Though synthetic pesticides are being used to reduce or prevent the harmful pests, they can pose serious problems to crop plants. Insects are much more closely related to man and other mammals than either plants or fungi and consequently it is perhaps not surprising that the greatest
threat to the environment has come from the large scale application of synthetic insecticides rather than from fungicides and herbicides. Some of the disadvantages of synthetic pesticides are as follows:

(i) They should be used with caution, since they are poisonous or toxic to men and mammals.

(ii) They may be irritating to skin, eyes and nose.

(iii) Pesticides containing mercury compounds when come in contact with skin, cause serious injury.

(iv) Excessive use of synthetic pesticides kills the phloem cells and this will prevent translocation due to which the crop plant may die.

(v) Due to constant use of certain group of pesticides, certain pests develop resistance to these and thus resistant strains of pests develop.

(vi) Some other hazards of synthetic pesticides are ground and surface water contamination, damage to soil-microorganism and negative effect on plant bio-diversity.\(^{14}\)

A multicentric questionnarie based study revealed that synthetic pesticides are harmful to human health and their use should be avoided and discouraged. The people who use these pesticides for extended periods may suffer neurotoxic and immunotoxic hazards. Hence, the pesticides which are used, should be safe to human health, wild life and non-target species.

So, there will be increasing emphasis on integrated biological and chemical pest control methods to reduce the use of chemicals, which will mean less dangers of environmental pollution and less opportunity for the emergence of resistant strains of pests. The food-environmental challenge reflects concern for providing environmentally acceptable solutions for weed, pest and disease control in the face of a rapidly expanding global population.\(^{15}\)
In conclusion, there appears little prospect in the foreseeable future that biological control measures, such as, the introduction of resistant crop varieties, cultural control, genetic methods or the use of natural products as pesticide, will replace chemical pesticides from their dominant position. Plants are good indicators as well as mitigators of environmental pollution. They can play an elegant role in probing such environmental attributes that can be used in monitoring pollution caused by synthetic pesticides.\textsuperscript{16}

Thus the pesticides will need to be highly potent, target pest specific and harmless to the environment. To make these features possible natural products and bio-pesticides are being increasingly explored. The pesticidal chemical must have minimum ecological effects, if it is to be a commercially viable pesticide in the modern pollution-conscious society.\textsuperscript{17}

**NATURAL PESTICIDES FROM PLANTS**

Continued search\textsuperscript{18-41} of compounds concerning to pesticidal activity isolated from plants has revealed that many compounds which are extracted from plants and possess pesticidal activity\textsuperscript{42-56} are used to kill the pests. Plants are the good source of well-established traditional pesticides. For example—plant extract of tobacco (*Nicotiana tabacum*), pyrethrum (*Chrysanthemum cinerariaefolium*), derris (*Derris elliptica*), red squill (*Urginea maritima*) and nux-vomica (*Strychnos nux-vomica*) are widely and commonly used for this purpose.\textsuperscript{57-70}

A deep sweep in the available literature on pesticidal compounds of plant origin has revealed that some recent pesticidal compounds, which have established their reputation, have been isolated from Indian Medicinal Plants, which are given below in table–1.1.
<table>
<thead>
<tr>
<th>S.No.</th>
<th>Name of the Plant</th>
<th>Part of the plant</th>
<th>Isolated compounds</th>
<th>Structure</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td><em>Pyrethrum cinerariaefolium</em></td>
<td>Flower</td>
<td>Pyrethrin-1</td>
<td>I</td>
<td>71</td>
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<tr>
<td>2.</td>
<td><em>Strychnos nux vomica</em></td>
<td>Seeds</td>
<td>Strychnine</td>
<td>II</td>
<td>72</td>
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<tr>
<td>3.</td>
<td><em>Derris elliptica</em></td>
<td>Roots</td>
<td>Rotenone</td>
<td>III</td>
<td>73</td>
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<tr>
<td>4.</td>
<td><em>Nicotiana tobaccum</em></td>
<td>Leaves</td>
<td>Nicotine</td>
<td>IV</td>
<td>74</td>
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<tr>
<td>5.</td>
<td><em>Artemisia annua</em></td>
<td>Aerial part</td>
<td>Artemisinin</td>
<td>V</td>
<td>75</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Arteether</td>
<td></td>
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</tr>
<tr>
<td>6.</td>
<td><em>Terminalia bellerica</em></td>
<td>Fruits</td>
<td>Termilignan</td>
<td>VII</td>
<td>76</td>
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<td></td>
<td></td>
<td>Thannilignan</td>
<td>VIII</td>
<td></td>
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<tr>
<td>7.</td>
<td><em>Lantana Camara</em></td>
<td>Aerial part</td>
<td>Lantanoside</td>
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<td></td>
<td></td>
<td>Lantanone</td>
<td>X</td>
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<td></td>
<td>Linaroside</td>
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<td></td>
<td>Camarinic acid</td>
<td>XII</td>
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<td>8.</td>
<td><em>Aglaia oligophylla</em></td>
<td>Aerial part</td>
<td>Aglaroxin A</td>
<td>XIII</td>
<td>78</td>
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<td></td>
<td></td>
<td></td>
<td>3’methoxy aglaroxin A</td>
<td>XIV</td>
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<td></td>
<td></td>
<td></td>
<td>Aglain</td>
<td>XV</td>
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<td></td>
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<td></td>
<td>Aglaforbesin</td>
<td>XVI</td>
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<tr>
<td>9.</td>
<td><em>Pseudolarix Kaempferi</em></td>
<td>Roots and trunk bark</td>
<td>Psedolaric acid A</td>
<td>XVII</td>
<td>79</td>
</tr>
<tr>
<td>S.No.</td>
<td>Name of the Plant</td>
<td>Part of the plant</td>
<td>Isolated compounds</td>
<td>Structure</td>
<td>Reference</td>
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<tr>
<td>10.</td>
<td><em>Dinosperma erythrococca</em></td>
<td>Seeds</td>
<td>Erythroccamamide A</td>
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<td></td>
<td>Erythroccamamide B</td>
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<td></td>
<td>Erythroccamamide C</td>
<td>XX</td>
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<td></td>
<td></td>
<td></td>
<td>N (2-hydroxy 2methylpropyl) 6-phenyl 2,4 hexadienamide</td>
<td>XXI</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>N(2methyl propyl)6-phenyl 2,4 hexadienamide</td>
<td>XXII</td>
<td></td>
</tr>
<tr>
<td>11.</td>
<td><em>Phenax angustifolius</em></td>
<td>Leaves</td>
<td>2 hydroxy 2 (3',4' dihydroxy-phenyl) methyl 3-(3'',4'' di-methoxyphenyl) methyl-γ-</td>
<td>XXIII</td>
<td>81</td>
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<td></td>
<td></td>
<td></td>
<td>butyrolactone</td>
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<td></td>
<td></td>
<td>2-hydroxy 2 (4'--O-β-D glucopyra-nosyl-3' hydroxy-phenyl) methyl-3 (3'', 4'' dimethoxy</td>
<td>XXIV</td>
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<td></td>
<td></td>
<td></td>
<td>phenyl) methyl-γ-butyrolactone</td>
<td></td>
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<tr>
<td>12.</td>
<td><em>Celastrus angulatus</em></td>
<td>Root-bark</td>
<td>Celangulin II</td>
<td>XXV</td>
<td>82</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Celangulin III</td>
<td>XXVI</td>
<td></td>
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<td></td>
<td>Celangulin IV</td>
<td>XXVII</td>
<td></td>
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<tr>
<td>13.</td>
<td><em>Neorautanenia mitis</em></td>
<td>Roots</td>
<td>Neoteneone</td>
<td>XXVIII</td>
<td>83</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>Dolineone</td>
<td>XXIX</td>
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<td></td>
<td>Pachyrrhizine</td>
<td>XXX</td>
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<td>Neoduline</td>
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<td>No.</td>
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<tr>
<td>1</td>
<td>Pyrethrin-1</td>
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<tr>
<td>2</td>
<td>Strychnine</td>
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<tr>
<td>3</td>
<td>Rotenone</td>
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<tr>
<td>4</td>
<td>Nicotine</td>
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<tr>
<td>5</td>
<td>Artemisinin</td>
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</table>
[12] Camarinic acid

[13] Aglaroxin A

[14] 3'-methoxy aglaroxin A

[15] Aglain
[21] N-(2-methyl propyl) 6-phenyl 2,4 hexadienamide

[22] N-(2-methyl propyl) 6-phenyl 2,4 hexadienamide

[23] 2-hydroxy-2-(3′,4′ dihydroxy phenyl) methyl-3-(3″,4″ dimethoxy phenyl) methyl γ butyrolactone

[24] 2-hydroxy-2-(4′-O-b-D-glucopyranosyl-3′hydroxy phenyl) methyl-3-(3″,4″ dimethoxy phenyl) methyl γ butyrolactone
[25] Celangulin II

[26] Celangulin III

[27] Celangulin IV
[28] Neotenone

[29] Dolineone

[30] Pachyrrhizine

[31] Neoduline
Above deliberations reveal that a large number of compounds have been isolated from different parts of plants of different families as tabulated (table 1.1) above and as such there exists enough justification to stimulate curiosity to carry out further systematic phytochemical investigation with a view to isolate, purify and elucidate the structures of pesticidal compounds by using modern spectroscopic techniques. As such the following plants were selected and identified by the authoress for the present investigation in view of their significant pesticidal activity.

The description of the plants are given below:-

1. **Nerium indicum** Linn.

   *Nerium indicum* Linn.\(^{84-93}\) belongs to natural order Apocyanaeae and is commonly known as 'Kaner' in Hindi. It is extensively cultivated throughout the greater parts of India.

   The plant is medicinally important. Its root is a powerful attuivant and the paste of the roots with water is applied to chancre and ulcers. Extract of its leaves is used to reduce swellings. The oil obtained from its root bark is used in the treatment of skin diseases and leprosy. (Photograph-1)

2. **Tridax procumbens** Linn.

   *Tridax procumbens* Linn.\(^{94-104}\) belongs to natural order Compositae (Asteraceae) and is commonly known as 'Ghamra' in Hindi.

   The leaves of the plant are used in the treatment of bronchial catarrh, dysentery, diarrhoea and for restoring hairs. Leaf juice possesses anti septic, insecticidal and parasiticidal properties. It is also used to check hemorrhage from cuts, bruises and wounds. (Photograph-2)

   The compounds isolated from the plants *Nerium indicum* Linn. and *Tridax procumbens* Linn. are displayed in table-1.2.
Photograph 1: Flowers Nerium indicum Linn.
Photograph 2: Flowers Tridax procumbens Linn.
<table>
<thead>
<tr>
<th>S.No.</th>
<th>Name of the plant</th>
<th>Part of the plant</th>
<th>Isolated Compounds</th>
<th>Structure</th>
<th>Reference</th>
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<td>Isoquercetin</td>
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[1] Kanerocin

[2] Neriucoumaric acid

[3] Isoneriucoumaric acid


[5] Uvaol

[12] Gentiobiosyl beaumontoside

[13] Cerdollagenin-3-\(\beta\)-neritrioside
[14] Oleandrigenin-β-D-glucoside


[16] Oleandrigenin-β-odorotrioside

[17] Digitoxigenin-β-neritrioside
[18] Adynerigenin-\( \beta \)-neritriose

[19] Oleandriegenin-\( \alpha \)-oleabioside

[20] Oleandriegenin-\( \beta \)-neribioside

[21] Digitoxigenin-\( \beta \)-D-glucoside
[22] Oleandrigenin-β-neritrioside

[23] Glucosyl chryoptograndoside


[25] Dexamethasone

[26] Fumaric acid
[27] β-Sitosterol

[28] Luteolin

[29] Glucoluteolin

[30] Quercetin

[31] Isoquercetin
CHAPTER-I

PROBLEM TAKEN AND WORK DONE:

From the time immemorial human beings have been depended on plants as a source of food and medicines. Ever since the dawn of civilization man has continually endeavoured to improve his living conditions; in his efforts to produce adequate supplies of food man has been opposed by the ravages wrought by insects, pests and crop diseases. The major pests inhibiting the growth of agricultural crops are insects, fungi and weeds and the idea of combating these pests by the use of chemicals is not new.\textsuperscript{116} The idea of the internal treatment of plants with chemicals (plant chemotherapy) goes back at least to the twelfth century when various substances such as spices, colouring matters and medicines were inserted into the bore holes of fruit trees to endeavour to improve the fruit.\textsuperscript{117,118} Thus the use of pesticidal chemicals in agriculture makes it possible to save approximately more than one third of our crops. But they can also pose serious problem to the plant life.\textsuperscript{119,120}

Since last 60 years pesticides are transmitted through water, air and soil in the environment. Through air pesticides residues deposit on crops. Pesticides also find way into over ground water bodies thus affect aquatic life and pollute water for a long time.\textsuperscript{121,122}

So there is a need to search and develop new natural pesticides which are active at highly acceptable levels, biodegradable and do not leave toxic residues.

Also from the ancient times a number of natural pesticides have been used to destroy the pests. Some of these are still in considerable use in the countryside e.g. Pyrethrum from \textit{Chrysanthemum cinarariaefolium}, Rotenone from \textit{Derris} species, Artemisinin from \textit{Artemisia annua},

_____________________

Department of Chemistry, Dr. H.S. Gour University Sagar
Strychnine from Strychnos nux vomica, Nicotine from Nicotiana tabaccum and Azadirachtin from Azadirachta indica. From very old ages throughout the world man has tried to mitigate pests by making proper use of natural pesticidal agents of plants origin but some of the useful plants possessing pesticidal agents are yet to be studied properly by research workers.

The war against pests through the development of new pesticides is an ongoing continuous process to counter the alarming global increase in the prevalence of different types of pests and damages caused by them particularly in developing countries like India.

Therefore, it was thought worthwhile by the authoress to take up the challenging task of unrevealing the secret of the pesticidal values of the indigenous plants; (1) Nerium indicum Linn. and (2) Tridax procumbens Linn. by investigating them phytochemically and the findings of the authoress are summerised below :-

[I] Isolation and characterization of the novel triterpenoidal saponin; Kanerocin 3-O-β-D-glucopyranosyl (1→4) O-α-L-arabinopyranosyl (28→1)-β-D-glucopyranosyl ester from the flowers of Nerium indicum Linn.

This chapter deals with the isolation and study of triterpenoidal saponin AS-I (yield 0.1566%), molecular formula $C_{x}H_{y}O_{z}$, m.p. 270-271°C and [M$^+$] 910 (FABMS). It was obtained by column chromatography of benzene soluble fraction of the concentrated ethanolic extract of the flowers of Nerium indicum Linn. Compound AS-I showed significant absorption bands in the IR spectrum at $\nu_{\text{KBr}}^{\text{max}}$ cm$^{-1}$: 3377.8 (–OH), 1725.1 (ester –C=O stre), 1629.6 (C=C), 2880.5 (C-H stre),
1370.2 (C-H bending), 1367.5, 1320.4 (triterpenoidal nature) and 867.2 (cyclohexane ring). It's $^1$H-NMR signals were recorded at (300 MHz, DMSO): $\delta$ 0.37 (3H, s, H-23 Me), $\delta$ 0.83 (3H, s, H-24 Me), $\delta$ 0.87 (3H, s, H-25 Me), $\delta$ 0.90 (3H, s, H-26 Me), $\delta$ 0.92 (3H, s, H-27 Me), $\delta$ 1.59 (3H, s, H-29 Me), $\delta$ 1.67 (3H, s, H-30 Me), $\delta$ 1.24 (2H, m, H-1), $\delta$ 1.90 (2H, m, H-2), $\delta$ 3.69 (1H, t, H-3), $\delta$ 1.63 (1H, m, H-5), $\delta$ 1.45 (2H, m, H-6), $\delta$ 1.32 (2H, m, H-7), $\delta$ 1.54 (1H, m, H-9), $\delta$ 2.0 (2H, m, H-11), $\delta$ 1.20 (2H, m, H-12), $\delta$ 2.40 (1H, t, H-13), $\delta$ 1.14 (2H, m, H-15), $\delta$ 1.04 (2H, m, H-16), $\delta$ 5.23 (1H, t, H-21), $\delta$ 1.71 (2H, m, H-22), $\delta$ 6.33 (1H, d, J= 6.0 Hz, H-1' arabinose anomic proton ), $\delta$ 5.23 (1H, d, J = 7.0 Hz, H-1'' glucose anomic proton), $\delta$ 6.23 (1H, d, J = 6.0 Hz, H-1''' glucose anomic proton), $\delta$ 4.20-4.60 (4H, m, arabinose protons), $\delta$ 4.08-4.18 (5H, m, glucose$^2$ protons), $\delta$ 3.82-4.06 (5H, m, glucose$^3$ protons) and $^{13}$C-NMR signals were recorded at (300 MHz, CDCl$_3$): $\delta$ 39.8 (C-1), $\delta$ 26.6 (C-2), $\delta$ 91.6 (C-3), $\delta$ 40.3 (C-4), $\delta$ 56.2 (C-5), $\delta$ 19.3 (C-6), $\delta$ 32.2 (C-7), $\delta$ 42.0 (C-8), $\delta$ 61.8 (C-9), $\delta$ 38.0 (C-10), $\delta$ 22.2 (C-11), $\delta$ 31.0 (C-12), $\delta$ 40.1 (C-13), $\delta$ 42.8 (C-14), $\delta$ 28.8 (C-15), $\delta$ 33.5 (C-16), $\delta$ 50.5 (C-17), $\delta$ 127.7 (C-18), $\delta$ 138.5 (C-19), $\delta$ 141.8 (C-20), $\delta$ 118.2 (C-21), $\delta$ 38.7 (C-22), $\delta$ 16.7 (C-23), $\delta$ 28.7 (C-24), $\delta$ 16.4 (C-25), $\delta$ 16.9 (C-26), $\delta$ 15.2 (C-27), $\delta$ 176.3 (C-28), $\delta$ 23.4 (C-29), $\delta$ 20.7 (C-30), $\delta$ 105.6 (C-1'), $\delta$ 76.8(C-2'), $\delta$ 72.5 (C-3'), $\delta$ 67.3 (C-4'), $\delta$ 63.1 (C-5'), $\delta$ 92.8 (C-1''), $\delta$ 79.5 (C-2''), $\delta$ 77.3 (C-3''), $\delta$ 70.5 (C-4''), $\delta$ 78.0 (C-5''), $\delta$ 61.1 (C-6''), $\delta$ 105.6 (C-1''''), $\delta$ 79.4 (C-2'''), $\delta$ 76.8 (C-3'''), $\delta$ 73.5 (C-4''''), $\delta$ 76.5 (C-5'''), and $\delta$ 70.7 (C-6'''').

Its FABMS showed significant fragmentation peaks m/z 910 [M$^+$], 586, 454, 437, 436, 409, 246, 208, 201, 190 and 189.
On acid hydrolysis AS-I yielded sapogenin AS-I (A) which was identified as Kanerocin (by superimposable spectral analysis). The sugars obtained from the acid hydrolysis of the saponin AS-I were identified as L-rhamnose (R_f 0.22) and D-glucose (R_f 0.18) by CoPC and CoTLC, whereas periodate oxidation, partial and enzymatic hydrolysis showed the bisdesmosidic nature of the glycoside and sugars were present in the ratio of 2:1. One glucose unit was linked through –COOH group at C-17 of sapogenin by β-glycosidic linkage and one glucose and one arabinose units were linked to the sapogenin through 3-O-glycosidic linkage. D-glucose was terminal sugar and L-arabinose was linked to sapogenin via α-linkage and D-glucose was linked to L-arabinose via β-linkage.

Thus the triterpinoidal saponin AS-I was identified as; Kanerocin 3-O-β-D-glucopyranosyl (1→4)-O-α-L-arabinopyranosyl (28→1)-β-D-glucopyranosyl ester.

![Chemical Structure of AS-I](image)

(II) Isolation and study of the cardenolide; Oleandrinogenin-3-O-α-L-rhamnopyranoside from the flowers of Nerium indicum Linn.

This chapter incorporates the study of the cardenolide AS-II (yield 0.7166%), molecular formula C_{51}H_{86}O_{10}, m.p. 178-180°C and [M'] 578 (FABMS), which was obtained by column chromatography of the ethyl
acetate soluble fraction of the concentrated ethanolic extract of the flowers of *Nerium indicum* Linn. Cardenolide AS-II showed significant absorption bands in the IR spectrum at $\nu_{\text{max}}^{\text{KBr}} \text{ cm}^{-1}$: 3487.0 (–OH), 2928.0 (C–H asymmetric stret.), 1794, 1735 (α, β unsaturated lactone ring), 1663.0 (C = C stret.), 1460.0 (C–H asymmetric bending), 1380.6 (C–H symmetric bending), 1345, 1310, 1211.9, (C–H twisting and wagging), 1075.3 (C–O stret.) and 1185.3 (C–<sub>O</sub> stret.). It's ¹H-NMR signals were recorded at (300 MHz, DMSO): δ 0.93 (3H, s, H-18 Me), δ 0.96 (3H, s, H-19 Me), δ 1.94 (3H, s, –OCOCH₃), δ 1.68 (2H, m, H-1), δ 1.89 (2H, m, H-2), δ 4.05 (1H, m, H-3), δ 1.46 (2H, m, H-4), δ 1.75 (1H, m, H-5), δ 1.15 (2H, m, H-6), δ 1.78 (1H, m, H-9), δ 1.40 (2H, m, H-11), δ 2.10 (2H, m, H-12), δ 2.56 (1H, d, J= 3.1 Hz, H-15), δ 5.45 (1H, t, H-16), δ 2.82 (1H, m, H-17), δ 4.85 (2H, s, H-21), δ 5.80 (1H, s, H-22), δ 5.54 (1H, d, J= 8.0 Hz, H-1' anomic proton), δ 1.35 (3H, complex signal, CH₃ rhamnose) δ 2.40 (4H, m, rhamnose protons) and ¹³C-NMR signals were recorded at (300 MHz, CDCl₃): δ 27.60 (C-1), δ 28.47 (C-2), δ 73.66 (C-3), δ 116.72 (C-4), δ 138.4 (C-5), δ 28.74 (C-6), δ 26.75 (C-7), δ 32.37 (C-8), δ 48.93 (C-9), δ 58.42 (C-10), δ 21.46 (C-11), δ 31.70 (C-12), δ 49.44 (C-13), δ 83.48 (C-14), δ 28.58 (C-15), δ 78.51 (C-16), δ 49.12 (C-17), δ 15.76 (C-18), δ 14.83 (C-19), δ 176.25 (C-20), δ 73.76 (C-21), δ 126.54 (C-22), δ 102.4 (C-1'), δ 70.4 (C-2'), δ 70.9 (C-3'), δ 71.8 (C-4'), δ 71.0 (C-5') and δ 17.5 (C-6'). It's mass spectrum showed significant fragmentation peaks at m/z 578 [M⁺], 432, 372, 354, 336, 284, 204, 162, 148 and 133.

On acid hydrolysis AS-II yielded cardiogenin AS-II (A) which was identified as oleandrogenin by mixed melting point and superimposable spectral analysis. The sugar obtained by acid hydrolysis of AS-II was identified as L-rhamnose (R₉ 0.36) by CoPC and CoTLC.
Periodate oxidation, acid and enzymatic hydrolysis of AS-II showed that the cardiogenin and sugar were present in equimolar ratio and L-rhamnose was linked to oleandrogenin via α-linkage.

Thus the cardenolide AS-II was identified as; Oleandrogenin-3-O-α-L-rhamnopyranoside.

(III) Isolation and structural study of steroidal saponin; β-Sitosterol-3-O-β-D-xylopyranoside from the flowers of *Tridax Procumbens* Linn.

This chapter deals with the study of the steroidal saponin AS-III (yield 0.078%), molecular formula $C_{34}H_{58}O_{9}$, m.p. 196-198°C and $[M^+]$ 546 (FABMS), which was obtained by column chromatography of the methanol soluble fraction of the concentrated Ethanolic extract of the flowers of *Tridax procumbens* Linn. Its IR spectrum showed absorption bands at $V_{\text{max}}^{\text{KBr}}$ cm$^{-1}$: 3425.0 (–OH), 2940.0 (CH$_3$–CH$_2$ stre.), 1650.3 (C=C stre.), 1470.0 (C–H bending), 1367.7 (–CH(12)H stre.), 2928.0 (–CH$_3$ stre.), 1390, 1305, 1060.5, 1025.0 (twisting and wagging of C-H bond in steroidal nucleus), 988, 960, 892 (cyclohexane ring), 825, 800 (C=C deformation) and 1H-NMR signals were recorded at (300 MHz, DMSO):
δ 1.02 (2H, m, H-1), δ 1.36 (2H, m, H-2), δ 3.79 (1H, m, H-3), δ 2.64 (2H, m, H-4), δ 5.30 (1H, t, H-6), δ 1.97 (2H, m, H-7), δ 1.52 (1H, m, H-8), δ 0.96 (1H, m, H-9), δ 1.45 (2H, m, H-11), δ 1.71 (2H, m, H-12), δ 1.12 (1H, m, H-14), δ 1.54 (2H, m, H-15), δ 4.60 (2H, m, H-16), δ 1.73 (1H, m, H-17), δ 0.68 (3H, s, H-18 Me), δ 0.99 (3H, s, H-19 Me), δ 1.92 (1H, m, H-20), δ 0.90 (3H, d, J=3.0 Hz, H-21 Me), δ 1.63 (2H, m, H-22), δ 1.66 (2H, m, H-23), δ 1.57 (2H, m, H-24), δ 1.55 (1H, m, H-25), δ 0.83 (3H, d, J= 6.9 Hz, H-26 Me), δ 0.81 (3H, d, J=6.9 Hz, H-27 Me), δ 1.50 (2H, m, H-28), δ 0.85 (3H,t, H-29 Me), δ 3.35 (1H, d, J=3.4 Hz, H-1' anomeric proton), δ 3.12-3.30 (6H, m, xylose protons). Its 13C NMR signals were recorded at (300 MHz, CDCl3); δ 36.9 (C-1), δ 30.0 (C-2), δ 79.5 (C-3), δ 38.6 (C-4), δ 140.1 (C-5), δ 121.8 (C-6), δ 31.8 (C-7), δ 32.3 (C-8), δ 50.4 (C-9), δ 36.5(C-10), δ 21.2 (C-11), δ 39.4 (C-12), δ 42.0 (C-13), δ 56.5 (C-14), δ 24.3 (C-15), δ 28.0 (C-16), δ 55.8 (C-17), δ 11.5 (C-18), δ 19.0 (C-19), δ 36.2 (C-20), δ 18.5 (C-21), δ 34.1 (C-22), δ 26.2 (C-23), δ 45.8 (C-24), δ 29.3 (C-25), δ 19.4 (C-26), δ 18.8 (C-27), δ 23.2 (C-28), δ 11.9 (C-29), δ 107.2 (C-1'), δ 75.5 (C-2'), δ 78.3 (C-3'), δ 74.5 (C-4') and δ 65.4 (C-5').

It's FABMS showed significant fragmentation peaks at m/z 414 [M'] 399, 396, 381, 329, 303, 301, 273, 271, 255, 231, 213 and 139.

On acid hydrolysis AS-III yielded aglycone identified as β-sitosterol (m.m.p. and superimposable spectra with authentic sample) and D-xylose (Rₐ 0.20) as sugar moiety (CoPC and CoTLC).

Periodate oxidation, acid and enzymatic hydrolysis of AS-III showed that the aglycone and sugar were present in equimolar ratio and D-xylose was linked to β-sitosterol via α-linkage.
Thus the steroidal saponin AS-III was identified as \( \beta \)-Sitosterol 3-O-\( \beta \)-D-xylopyranoside.

[IV] This chapter has been divided into three parts:

PART (A): Isolation and structural study of the isoflavonoidal glycoside; Isoquercetin 7-O-\( \beta \)-D-glucopyranoside from the flowers of *Tridax procumbens* Linn.

This part deals with the isolation and study of isoflavonoidal glycoside AS-IV (yield 0.0993%), molecular formula \( \text{C}_{21}\text{H}_{20}\text{O}_{13} \), m.p. 226-228°C and \([M^+]\) 464 (FABMS), which was obtained by column chromatography of ethyl acetate soluble fraction of the concentrated ethanolic extract of the flowers of *Tridax procumbens* Linn. Glycoside AS-IV showed significant bands in the UV spectrum at \( \lambda_{\text{max}}^\text{MeOH} \): 258, 365 nm; \( \lambda_{\text{max}}^{\text{AcOH}} \): 240, 410 nm; \( \lambda_{\text{max}}^{\text{AcCl}+\text{HCl}} \): 355, 425 nm; \( \lambda_{\text{max}}^{\text{NaOMe}} \): 320, 412 nm; \( \lambda_{\text{max}}^{\text{NaOAc}} \): 270, 325 nm; \( \lambda_{\text{max}}^{\text{NaOAc}+\text{H}_2\text{BO}_3} \): 265, 385 nm. It’s IR spectrum showed significant bands at \( v_{\text{KBr}}^{\text{max}} \text{ cm}^{-1} \): 3390.4 (-OH), 2936.7 (C-H strect.), 1640.8 (>C=O strect.), 1610.4 (aromatic ring system), 1387.0 (C-O-C bending), 1050.6 (C-O-C strect.), 845.8 (Two adjacent H- atom in ring system) and \(^1\text{H}-\text{NMR}\)
signals were recorded at (300MHz, DMSO) : δ 7.62 (1H, s, H-2), δ 7.49 (1H, d, J=7.8 Hz, H-6), δ 6.71 (1H, d, J=7.9 Hz, H-5), δ 6.35 (1H, d, J=2.1 Hz, H-8), δ 6.30 (1H, d, J=2.0 Hz, H-6), δ 5.15 (1H, d, J=8.0 Hz, H-1") anomic proton), δ 3.50 (6H, complex signal glucose protons). Its \textsuperscript{13}C-NMR spectrum showed signals at (300 MHz, CDCl\textsubscript{3}) δ 133.5 (C-2), δ 156-4 (C-3), δ 177.0 (C-4), δ 160.5 (C-5), δ 98.6 (C-6), δ 164.2 (C-7), δ 92.8 (C-8), δ 156.2 (C-9), δ 103.8 (C-10), δ 120.5 (C-1'), δ 115.3 (C-2'), δ 146.4 (C-3'), δ 147.7(C-4'), δ 116.0 (C-5'), δ 120.2 (C-6'), δ 90.0 (C-1'"), δ 73.2 (C-2'"), δ 7.5 (C-3"), δ 69.9 (C-4"), δ 76.0 (C-5"), δ 66.8 (C-6") and important fragmentation peaks in the FABMS were recorded at m/z: 464 [M\textsuperscript{+}], 302, 274, 153, 152, 151 and 124.

On acid hydrolysis AS-IV gave aglycone AS-IV (A) identified as isoquercetin and sugar moiety, which was identified as D-glucose (R\textsubscript{f} 0.20) by CoPC and CoTLC.

Periodate oxidation, acid and enzymatic hydrolysis of AS-IV showed that the aglycone and sugar were present in equimolar ratio and D-glucose was linked to aglycone via β-linkage.

Thus the glycoside AS-IV was identified as; Isoquercetin 7-O-β-D-glucopyranoside.
PART (B): Studies on the antifungal activity of the compounds AS-I, AS-II, AS-III and AS-IV

This part describes the study on antifungal activity of the compounds isolated from indigenous pesticidal plant. Compounds AS-I and AS-II were isolated from plant *Nerium indicum* Linn. (N.O. Apocyanaceae) and compounds AS-III and AS-IV isolated from plant *Tridax procumbens* Linn. (N.O. Compositae). All the isolated compounds AS-I, AS-II, AS-III and AS-IV were screened for their antifungal activity by filter paper disc method. DMF was used as solvent whereas griseofulvin was used as standard antifungal agent.

The compounds AS-I and AS-II showed significant zone of inhibition against *Aspergillus niger* and *Fusarium oxysporium*. The compound AS-III was found to be more active against *Fusarium oxysporium*, *Penicillium digitatum* and *Trichoderma viride* while compound AS-IV was found to be effective against *Fusarium oxysporium* and *Penicillium digitatum*.

PART (C): Studies on the insecticidal screening of the compounds AS-I, AS-II, AS-III and AS-IV

This part describes the insecticidal screening of all the isolated compounds AS-I, AS-II, AS-III and AS-IV. The insecticidal activity was carried out by residual film method. Two common insect pests *Sitophilus oryzae* and *Periplaneta americana* were used as test insects to test the insecticidal activity. The natural insecticidal agents AS-I, AS-II and AS-III produced significant knockdown activity while compound AS-IV was found to be less active against both the insect pests.
REFERENCES


