PRODUCTION OF INSECT REPELLANT FROM MATERIALS SOURCED LOCALLY

BY

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A RESEARCH PROJECT REPORT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY FEDERAL UNIVERSITY OF TECHNOLOGY MINNA NIGER STATE.

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(B. ENG)

DEGREE IN CHEMICAL ENGINEERING.

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DECLARATION

I hereby declare that this project is my original work and has never to my knowledge been submitted elsewhere.

OCHOR CHINOYEREM (93/3551) DATE

CERTIFICATION

This is to certify that this Project work "PRODUCTION OF INSECT REPELLANT FROM MATE-RIALS SOURCED LOCALLY" which I have found adequate both in scope and quality for the partial fulfillment of the requirement for the award of Bachelor of Engineering degree in Chemical Engineering was presented by OCHOR E. CHINOYEREM 93/3551 of CHEMICAL ENGINEER-ING DEPARTMENT, FEDERAL UNIVERSITY OF TECHNOLOGY MINNA.

MR OLUTOYE

DR ODIGURE H. O. D CHEMICAL ENGINEERING DATE

DATE

DATE

DEDICATION

This Project is dedicated to the black race, which has sadly refused to develop scientifically. It is also dedicated to black Scientist all over the world that has contributed in one way or the other to the development of mankind. The good work must continue.

Above all it is dedicated to Almighty God and to my Mom.

ACKNOWLEDGEMENT

With profound gratitude to God for having seen me through and giving me the grace to get to this point in life.

I wish to express my sincere thanks to my supervisor Mr. *OLUTOYE* for his numerous advice. He adequately directed and assisted me. In this respect my special thanks goes to my H.O.D *Dr. O. ODIGURE* and the staff of Chemical Engineering Department.

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I am also idebted to the family of "Greatness" especially Greatness MOSES ABAYOWA.

And finally my heart goes out with big thanks to the two most wonderful, beatiful and exceptionally kind hearted people I have ever come across, they are my *MOM* and *DAD*. I will not have been able to write this project if not for them. I thank God for their lives.

OCHOR CHINOYEREM

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ABSTRACT

This Project work was done on Production of an Insect Repellant with emphasis on mosquitoes. Materials for the production were sourced locally. Citrus Sinuses Sawdust and Starch paste were used.

A lot of factors were considered and their effects on the efficiency of product looked into during the experimental research/production. Factors such as degree of drying of peels, drying temperature of peels and product, Particle size of pulverized material, and mixture mass ratio were researched into to know the optimum condition for good manufacturing and efficient functioning of product.

Tests were carried out on the products effectiveness on mosquitoes and they were quite satisfactory and it should be noted that health wise nothing was done to ascertain the possible health implication on using it.

CHAPTER ONE

1.0 INTRODUCTION

Repellants are chemical substances, which repel or keep off insects rather than killing them. They can be either vapour or contact type and both must induce the insect pest to move away from them and they must be acceptable to the host particularly if the host is a man, so that their application causes no discomfort.

There are two kinds of these insect repellants, which are the synthetic (artificially made) ones i.e. creosote and the naturally obtained ones such as Dimethylphthalate and deet. For the case under consideration, the source of the active ingredient is from a fruity plant citrus Sinensis (sweet orange) and the active ingredient is obtained from the peel and the ingredient is dimethylphthalate on ester (oil) with structural formular.



Boiling Point

On heating, it is transformed to the anhydrous from which on further heating decomposes and gives off an odour or smelly that keep off the insects.



Dimethylphthalate can be extracted from the peels by cold press and distillation of the resulting liquid or it can be used directly by drying the peels and grinding to powder.

1.1 AIM AND OBJECTIVE OF THIS PROJECT

The objective of this project is to produce an insect repellant using local material such as Sawdust and orange peels, thus minimizing cost and then source your raw materials locally from your immediate environment.

1.2 SCOPE OF WORK

This deals with the production of an insect repellant basically for mosquitoes. The active ingredient used Dimethylphthalate was sourced locally from the orange peel (sweet orange) and this was incorporated with sawdust and starch to produce a repellant such as a mosquito coil.

1.3 LIMITATIONS OF THE WORK

The Limitations enountered during this project work were the lack of information or recorded data on previous work done on this field by companies who carry out similar production of such product. These Companies refuse to make available these data, therefore there is almost no standard for comparing the work done on this project. Also the isolation of the active ingredient in the peels could not be achieved as a result of adequate equipment to facilitate this.

CHAPTER TWO

LITERATURE REVIEW

2.0 HISTORICAL DEVELOPMENT OF INSECT REPELLANT

Origin of insect repellant dates back to 176-147 BC when the 1st inscription on the temple walls of Horus at Edfie were believed to have been made by ancient Egyptian Priest. The Priest of the Temple of the Sun god (Rai) had used for hundred of years this repellant recipes, which were incorporated into incense which were offered to the gods and thus kept blood sucking insects away as the Priest did their duties in the temple.

One of the most celebrated Kings of Egypt was inscribed on the walls of the temple of Abu Sin bel offering such incense to their gods. Also remnants of such incense repellants were found in the tomb of Princess Hatter of the 13th Dynasty and are still on display in the museum at Gizah.

In the latter centuries, Arabs having raided the tombs of ancient Egypt and stole some of these recipes and improved on them. Their travelers who travelled long distance to trade or for slavery trade used these repellants a great deal.

Funny enough, the Myrians of ancient Mexico burnt dry cow dung incorporated with some ingredients as insect repellants. They also planted certain shrubs, which kept some unwanted insects away from their homes.

In 1705, Eliot a French Scientist produced the first refined form of insect repellant from citronella oil but citronella is too volatile and short lived to be an effective repellant. Men like Homer Pling improved on the work using various terpenes such as geramol, citronella and borneol, this deter mosquitoes from alighting or coming near objects coated with oil, but still citronella's high volatility was a problem. The advent of World War II caused the United States of America to embark on a major programme of screening chemicals for repellant properties.

The discovery of new repellant chemicals vital for the success of military operations against the Japanese in the far east. Indeed at one period, a soldier had a 90% chance of being attacked by a tropical disease before encountering the real enemy. By 1945, 7000 chemical compounds had been screened and in1952, the number had increased to 1100.

The initial tests were for insecticides and repellant properties against body lice, mosquito and chigger and latter house flies ticks and fleas were included.

Research in chemical repellants over the last 30 years or so has indeed been largely concentrated on the production of synthetic chemical. In early times, a number of herbal

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extracts had been recommended as insect repellants in particular citronella.

Dimethylphthalate, which became an important constituent of skin preparations but not effective against all species of insects blood sucking flies. During the war, other repellants were discovered, two of these were:

2 - 2 - dimethyl-6 - butyl carboxyl - 2 - 3 dihydro pyran-4 - one or indalone and 2 ehtylhexane 1 - 3 diol or Rutger 612, which were widely used together with dimethylphthalate to give a wide spectrum of repellant activity.

Deet N-N diethyl - in - toluamide super seeds all the above and is most widely used chemical repellant with a broad spectrum activity against mosquitoes flies chiggers and other biting insects.

2.1 SRUCTURES OF SOME REPELLANT COMPOUNDS MENTIONED ABOVE

Co, CH, Co, CH

Dimethylphthalate

Co, (CH,),

 $\begin{array}{c} \mathrm{CH}_3\text{-}\ \mathrm{CH}_2\text{-}\ \mathrm{CH}_2\text{-}\ \mathrm{CH}_2\text{-}\mathrm{CH}_2\text{-}\mathrm{CH}_2\text{-}\mathrm{CH}_2\text{-}\mathrm{CH}_3\\ & 1\\ \mathrm{OH}\ \ \mathrm{CH}_2\mathrm{OH}\end{array}$

22 dimethyl-6-butyl carbxyl -23 dihydropyran-4-one 2 ethylhexane - 1 - 3 diol orRutger 612

 $\operatorname{CON}\left(\mathrm{C_{2}H_{5}}\right)_{2}$ CH,

N - N diethyl in toluene

2.2 BIOCHEMICAL COMPOUNDS USED IN PRODUCTION OF REPELLANTS

These are usually extracts from plants and the most common ones are aromatic in structure, they include N - N diethyl in toluene 22dimethyl - 6 - butyl carboxyl 2 - 3 dihydropyram 4 - one and dimethylphthalate. There are others, which are not cyclic in structure such as Rutger 612 and even prythrum, which is usually used as an insect killer but can be sprayed with kerosene solution to act as repellant to insects.

2.3 HOW REPELLANTS WORK

Most insect repellants act by canceling out the attractive surface stimuli emitted by the host body at a distance. For instance, they prevent mosquitoes detecting a hot wet surface.

Some others tend to inhibit or cause the inhibitation of mitochondria electron transfer thus inhibiting oxidation linked with NADH. This reduces consumption depresses respiration and heartbeat causing the insect to move away within vicinity.

2.4 FORMS OF REPELLANT PRODUCTS

Different forms of repellant products have been produced or developed to suit different purposes and a majority of them have been found to be highly effective for such purposes. Such different forms are discussed below.

2.4.1 POWDERED FORM

Repellants produced in this form are in almost dust forms and are sprayed around the areas where the pest (insect) should be kept away from. Its action is by contact (the insect must come in contact with the material i.e. repellant powder). It is the contact type of repellant.

2.4.2 CREAM OR GEL

In this case, the repellant is produced as a semi liquid. They are usually applied to the skin and they either produce offensive odour to the insect or prevent the insect from having the normal feeling it gets when it is on it's host thus making the insect feel that the surface does not belong to its host.

2.4.3 OIL

Most repellants produced as oil are usually incorporated into other substances or materials. For example, there is a brand of paint that keeps off mosquitoes from a house when used to paint the house. The paint has oil repellant incorporated into it.

2.4.4 COMBUSTIBLE REPELLANT

The most common of these is the mosquito coil. They are produced like incense and on been lit by fire, they burn to give off fumes which keep off these unwanted insects. It is mainly referred to as the Vapour type.

CHAPTER THREE

3.1 EXPERIMENTAL PROCEDURE

Fresh peels of Citrus Sinensis peels were obtained and dried to remove moisture. This was done under atmospheric temperature and pressure 25°C and 760mmHg. And the resulting dry peels were pulverized into fine powder using a pulverizer. The resulting material was then passed through a series of sieve plate of different apertures to obtain a series of different particle sizes ranging 710 through 250um and less than 250um. Materials on each sieve plate were collected separately and weighed and each recorded against its particle sizes. The sawdust required was also passed through a sieve shaker and different particle sizes were obtained separately.

The starch paste used as binding agent to bind together both sawdust and grinded peels was made by dissolving starch powder first in cold water and latter with hot water at boiling point to form the starch paste. The hot starch produced was allowed to cool to room temperature to avoid damaging the structural and chemical composition of the active ingredient at high temperatures.

Now a ratio of 2:1 by mass of active ingredient to the sawdust was mixed and the cold binding agent was applied in small quantities until the binding properties come into play. Excess of the binding agent will make the mixture too watery to stick together. Note that the mixture of the active ingredient and the sawdust were done for the same particle size. When the desired state of the mixture is acquired, the mixture was then moulded into oblong shapes for ease of burning and these were dried under normal temperature and pressure 25°C and 760mmHg

3.2 EQUIPMENT USED FOR THE EXPERIMENT3.1.1 SIEVE SHAKER

This equipment is made of a series of sieve plates with different sieve apertures, which are arranged so that the aperture sizes of the plates increase from the bottom sets of plates to the top ones. This equipment was used to obtain fine sized particles of sawdust and the active ingredient, which was used in the production process (particle sizes below 250ium was preferable). The sawdust material to be separated into different particles sizes was poured into the top sieve plate and the set of plates were fixed in fitted on to the shaker and this was switched on for 2mins. After that the sieve plates were opened and the sawdust particles and each plate was collected according to particle sizes – the same was done for the grinded peels.

3.2.1 WEIGHING MACHINE

This is an electrically operated digital display-weighing machine. It was used to weigh and obtain the appropriate masses of the sawdust required and also that of the grinded peels needed for the production. The mass ratio should be 2:1 of the grinded peels to sawdust of both same particle sizes of below 250um.

3.1.3 MOULD

A mould is required to obtain the desired shape of the product, it should be made of carbon steel.

3.1.4 OVEN/DRYER

This equipment is used to dry the mixture of sawdust grinded peels and starch paste. It is an electrical powered dryer with a temperature regulator and the drying temperature should not exceed 25°C.

3.2 MATERIALS USED

3.2.1 CITRUS SINENSIS PEEL

These are obtained fresh from the fruity plant and they are dried under normal atmospheric temperature and pressure of 25°C. High temperature should not be used for drying to avoid damaging the active ingredient, which is an organic substance dimethylphthalate.

The dry peels are grinded into fine powder and sieved to obtain the finest of the particles which is used (particle size required is 250um and below). The amount of these peels (mass) should be twice or more than the mass of the sawdust. This material provides the required repulsive smell, which repels the blood-sucking insects when it is burnt.

3.2.2 SAWDUST

These can be obtained from sawmills and are highly inflammable. They aid the burning of the active ingredient, which is not highly combustible. The sawdust obtained is sieved and the finest of the particles is used for the production (below 25um) more preferable. The quantity of the sawdust should be quite less than the active ingredient (1:2).

3.2.3 STARCH PASTE

This can be obtained in its powdered form from market. It is made into paste slurry as explained in the experimental procedure. It should not be watery and should be allowed to cool before it is used. It serves as a binding agent and binds the sawdust and the active ingredient together, serves and acts well for particle sizes less than 500inm. The larger the particle size the more difficult it is to bind them together.

CHAPTER FOUR

DISCUSSION

This chapter will be based on discussing most factors, which were noted during the experiment to have affected the production process or the test of the final product positively or negatively. This will enable who ever is interested in the production of this product have a knowledge of the optimum conditions required to obtain the best of the product being processed.

The discussion of this work in this chapter was based on mentioning the ultimate factors required for the production of good products following the method used, and discussing these factors. No comparison was made with already existing data of already madde similar product. This is due to the fact that companies involved in this kind of production do not easily give out informations on such things, such as materials used for production and optimum production conditions required.

4.0 DRYING AND DRYNESS OF PEELS

The peels have to dry for easy size reduction, if they are not dried well enough the size reduction of the peels will not yield the required fine particles desired, instead they will be in chunks. The drying of the peels has to be done in low temperature not more than 25°C so as not to damage the chemical composition of the active ingredient, which is an organic substance and decompose at high temperature.

The resultant mixture of starch paste sawdust and peel have to be dried at same temperature 25°C for same reasons. The product should be dry enough to allow complete combination when lit up.

4.1.1 SIZE REDUCTION

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The pulverization of the materials (sawdust and peels) given the required particle size desired, which is 250um and below 250um. This size particle was found out to be suitable in the sense that the binding agent used will not bind together well enough particle sizes larger than this particle size and this will result to the production of a poor product.

Also for complete combustion of the product particle size of 250um and below or the best particle sizes to achieve this anything above this will give or pose difficulty in burning as the particle will be in chunks particles.

4.1.2 THICKNESS OF BINDING AGENT

The thickness of the paste matters a lot. If it is watery it will not serve the purpose well enough as it should. Thus it should not be watery or too thick so that the binding qualities will be effective.

4.1.3 QUANTITY OF PASTE APPLIED

This was found to vary depending on the thickness of the paste, if watery little of it is applied so that the product mixture does not become watery, if thick, more of it is applied to achieve the desired result.

It should be noted that the best way to achieve this effectively is to watch the thickness of the mixture as the binding agent is applied, and when the desired texture is attained you stop adding the paste.

4.1.4 MASS RATIO FOR THE MIXTURE

The effective mass ratio for he product mixture is 2:1, two of the grinded peels to one of the sawdust, and is even more effective if more of the active ingredients can be incorporated and less of the dust used. But the problem this might pose is that which arise from combustion of the end product. The active ingredient does not burn well on its own and need sawdust in reasonable amounts to aid its combustion. If more of the sawdust is used than the active ingredients then the repulsive odour or smell that should have been produced to repel insects is over shadowed by the smell of smoke, which results from the burning of the excess sawdust, and this reduces the effectiveness of the product.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.0 CONCLUSION

An effective blood-sucking insect repellant can be produced from the raw materials stated in this project work. The effectiveness of the product depends on sticking to the optimum conditions required for production.

Additives to broaden the scope of its effectiveness was looked into and even though not stated in this project work, as the research work on it was incomplete.

This insect repellant was tested and found effective but would be better and stronger with additives.

5.1 **RECOMMENDATION**

- I recommend that further extensive work should be done in this field especially concerning additives required to improve product.
- (2) Health implication of such products as this particular one should be looked into to know how safe it is to the user before the product can be recommended for human consumption.
- (3) I recommend the isolation of the active ingredient in the peel, this would produce a more effective product than using the whole peel.

TEST CARRIED OUT ON INSECTS

The test carried out on mosquitoes using the product showed that the mosquitoes start feeling the effect of the product after two minutes and soon become weak. Their recovery when this repellant is removed is faster than that of the mosquito coil sold in the market, which points to the facts that there is a need for an additive to prolong its effect and strength.

One possible additive which was look into is the wild variety of curry leaf Usilium Bacilicum. Which I dried and pulverized and added to saw-dust and the active increadent.

TEST FOR DIMETHYL PHTHLATE & SOME OF IT'S PROPERTIES

If isolation is done after cold press and distillation then the possible way to test for the present of Dimethyl phalate is to add sodium and diethyl acetate to the resulting liquid this then forms a complex compound which is whitish in colour and semi-solid in nature. This indicates the presence of Dimethyl phalate.

VISCOUSITY = 2.4 m/Nsm^{-2} BOILING POINT = 66.7° C SPECIFIC GRAVITY = 0.87THERMAL CONDUCTIVITY = $0.133 \text{ w/m}^{\circ}$ C AN HYDROUS FORM SUBLIMES AT 216° C

REFERENCES

JULIUS J. MEANN	(1983) Present Insecticide and Approaches to Discovery of		
	Environmentally Acceptable Chemical for Pests.		
KILMART WILSON	Introduction to Insects Pest Control.		
KUMAR	(1984) Insect Pest Control Edward Arnold Limited Oxford.		
MATTHEW G. H.	(1992) Pesticides Application Methods.		
MICHAEL GRAINGE and SALEEM AHMED			
	Handbook of Plants and their Pest Control Properties.		

SMITH J.B.

Dictionary for Organic Chemistry Compounds