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Detection of pesticide active ingredient into agricultural soil and carrot by using Fourier transform infrared spectroscopy (FTIR)

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Abstract
Pesticide is one of the best substances that are widely used in the agriculture field to control pest. Though pesticides are basically used to control pest but improper use of pesticide on crops can be harmful for the consumers. Carbendazim and Mancozeb+Metalaxyl are the active ingredient of Knowin and Ridomil gold and these pesticides were extensively used in Bangladesh for controlling pest and fungus. In this study the active ingredients of pesticide were detect from the soil and crops after harvesting by using QuEChERs method and sample were analyzed by Fourier Transform Infrared Spectroscopy (FTIR). By identifying and matching common functional group both of samples and pesticides at 1050 cm-1, 1450 cm-1, 1650 cm-1, 1840 cm-1, 2920 cm -1 and 3350 cm-1 intensity pesticides active ingredients were identified into samples. The results show that WHO class II pesticides are the most frequently used pesticides in the study area. Total 2 number of pesticide (1 Insecticide &1 Fungicide) were found into both soil and carrot samples. Food contamination is the biggest challenge for any developing country in the world like Bangladesh. It may be stated that the pesticide management techniques of the vegetable producers in the study area were inadequate. So, initiatives to improve farmers' pesticide understanding through increased training are critical and should be promoted by relevant stakeholders. Furthermore, authoritarian enforcement of pesticide sales restrictions should be implemented to raise farmers' ability on safety concerns.

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Keywords: Pesticides, Food Safety, FTIR

1. Introduction

By 2050, there will be an estimated 9 billion people on the planet, making the availability and accessibility of food more important challenges. Pesticides can help reduce output losses caused on by pests and provide food for the world's expanding population (Ali, *et al*., 2020) [3]. In order to enhance agricultural pest control and increase food production to meet the needs of the expanding global population, prudent pesticide usage is usually viewed as being imperative. To meet demand, it is crucial to use pesticides to safeguard crops during their growth, as well as during storage and transportation. Pesticide use worldwide is estimated to reach 2.5 million tons per year, and it is steadily increasing (FAO/WHO, 1996) [8]. Agriculture is commonly referred to as farming; it is an art and science that prudently endeavors to modify a portion of the Earth's crust via cultivation of plants and other crops as well as animal breeding for food or other requirements for human beings and economic benefit (Ismail, 2021) [11]. Modern farming increasingly requires the use of pesticides, which also significantly boosts agricultural output. But the widespread and careless use of pesticides is one of the largest problems with the environment and public health in the world today. The term "pesticide" refers to a wide range of chemicals, including insecticides, fungicides, herbicides, rodenticides, molluscicides, nematicides, plant growth regulators, and others (Aktar, Dwaipayan, & Chowdhury, 2009) [2].

Pesticides are chemicals that are both man-made and naturally occurring and are applied to eradicate pests including fungus, weeds, and insects that damage crops (Xiao, *et al*., 2010) [16]; (Clarke, Levy, Spurgeon, & Calvert, 1997) [6]; (Islam, Dastogeer, Hamim, Prodhan, & Ashrafuzzaman, 2014) [10]. Pesticide usage in Bangladesh, which was minimal until the year 1960, has dramatically increased in the last 50 years (Islam, *et al*., 2016) [9]. In a nation like Bangladesh, using pesticides to protect the crop from pests has become essential to maintaining and improving the current stage of harvest output (Islam, Dastogeer, Hamim, Prodhan, & Ashrafuzzaman, 2014) [10]. Farmers have used pesticides like sulphur and DDT to protect their crops since ancient times, which gives pest control a lengthy history. The adverse effects of these agents on consumers were quickly discovered, which led to the invention of additional agents like chlorinated insecticides (Akhtar, Yaqub, Hamid, Afzal, & Asghar, 2018) [1]. In fruits and vegetables, the three insecticides carbendazim, carbaryl, and thiabendazole are usually present. Chronic pesticide exposure also increases the risk of endocrine disruption, kidney and liver damage, neurological disorders, unexpected birth outcomes, and organ mutagenesis and carcinogenesis. Because of their small bodies, undeveloped immune systems, and fast development cycles, children are more vulnerable, particularly in the brain and neurological systems (Begum, Sultana, Ahmed, & Azad, 2019) [4]. In order to produce crops in a sustainable and high-quality manner and consume wholesome food, it is recommended that effective execution of government policies, ongoing oversight, expanding IPM methods, utilizing indigenous technology, and raising farmers' understanding of pesticide usage overall (Islam, *et al*., 2016) [9]. Major legislative initiatives for improving pesticide regulation and successful implementation, boosting farmers' awareness of the effects of pesticide usage, and extending IPM techniques are advised in order to safeguard poor farmers in their pursuit of agricultural livelihoods (Rahman, 2002) [14]. Fourier Transform Infrared Spectroscopy (FTIR) is one of the best analytical techniques that used for qualitative analysis of pesticide. By identifying functional group of active ingredients of pesticide then compare it soil and vegetables sample. This technique is best for this study because FTIR is highly advantageous as well as due to its rapid, nondestructive nature and finally minimal sample preparation requirements. The decoction of active ingradients of pesticide on soil and crops sample QuEChERS (Quick, Easy, Cheap, Effective, Rugged, and Safe) method is one of the best methods because of its efficiency and accuracy. Moreover, this method is very useful because of rapid and cost effective.

After gaining its independence, Bangladesh's economy was mostly driven by the agricultural sector. It provided about 60% of GDP. As we all know, Bangladesh's agriculture is essential for providing employment, a living, and contributing to the country's GDP. Over the past ten years, it has made less of a contribution, going from 17.6% in 2010 to 12.6% in 2020. The industry is essential to our economy, helping to alleviate poverty and provide food security. Nevertheless, the sector has maintained strong levels of profitability and productivity in spite of the population's continued growth, which is anticipated to rise from 147.6 million in 2010 to 164.7 million in 2020, as well as the effects of the pandemic and climate change. The perception and attitude of farmers toward the risk of pesticide exposure, as well as a lack of education and inadequate awareness of safe methods in pesticide usage, including storage, handling, and disposal, may all contribute to the aggravation of these risks. Higher levels of education provide pesticide users with better access to information and more understanding of the hazards connected with pesticides, as well as how to prevent exposure. The purpose of this study was to detect active ingredients into soil and secondly into carrot vegetable that was grown in same soil sample. The study will be a scope to ensure food safety, build awareness about the impact of pesticides on human health with farmers and consumers.

2. Method and materials

2.1 Study area

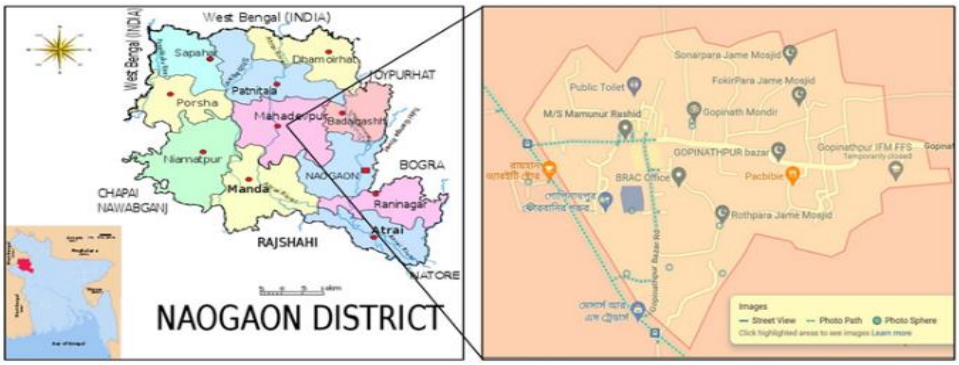

Fig 1: Map of study site

This study was carried out in near the Naogoan district in Bangladesh, which lies within latitudes from 24° 58' 0.55" **N** to 24° 57' 49.91"**N** and longitudes from 89° 5' 4.65"**E to** 89° 5' 11.76"**E** which is 500 km long (Fig. 1).

2.2 Samples and sampling procedure

The sampling was conducted within the winter season, spanning from December 2022 to January 2023. Three samples were obtained from each of the sampling sites and properly mixed together to create a composite sample from each site. A total of 0.5 kg of vegetable (Carrot) was randomly collected by three to four farmers and stored separately in specially labeled zip-lock polybags to the sample locations. Soil samples were gathered from the

vegetable farms using a plastic scooper at a depth of 0-30 cm. non-soil particles were eliminated, and the soil samples were thereafter placed in plastic containers that had been thoroughly cleaned beforehand and clearly marked with appropriate labels. The soil and vegetable samples were placed in a refrigerated container and promptly transferred, to the laboratory of the department of public health, Jahangirnagar University, for subsequent study.

2.3. Sample preparation

The process of sample preparation holds significant importance in the field of environmental analysis as it facilitates precise and dependable measurements of various substances like as pollutants, nutrients, and other analytes that are found in vegetables and soil. The selection of suitable methodologies is contingent upon the particular analytes, features of the matrix, and objectives of the analysis.

2.3.1. Vegetable & Soil

The vegetable & soil were subjected to an oven-drying process at a temperature of 105 °C. Subsequently, they were pulverized into a fine powder. Approximately 0.5 g of the resulting powdered sample was then introduced into the making of salt plate. Then sterilized all the equipment with acetone because of protection of cross contamination. A small amount of potassium bromide salt (KBr) generally one spatula has taken and also taken very tiny amount of sample. Then grind it properly with pestle. After making powder form sample has kept into base die and keep top die on it. Then set the die holder and put it under hydraulic press at 80 pressure and start the rotary vacuum pump for 2 minutes to remove the moisture. Then salt plat took out and package it with labeling and keep it into desiccator because of desiccator prohibit to grow moisture. The procedure of making salt plate of vegetables and soil were same. Then salt plate set into IR machine (Model no: IRPrestige-21) for analysis.

2.4 Analysis of sample

Infrared light is transmitted to the sample by means of a highly refractive prism; the angle at which the incoming infrared light strikes the sample is greater than the critical angle, which results in complete reflection. An infrared spectrum is obtained by measuring the amount of light that is completely reflected by the contact between the sample and the prism. The picture on the right illustrates how light passes through the sample's thin surface layer despite the term "total reflection." As a result, the spectrum produced is comparable to that obtained from a very thin slice of the same material.

3. Result and Discussion

3.1. Soil

FTIR Spectral Characteristic Analysis of agricultural soil and pesticide carbendazim were represent figure 3.1(a) as well as figure 3.1(b) shows FTIR spectra of another pesticide Mancozeb+Metalaxyl and soil samples. It can be observed from Fig. 3.1(a) that the spectral intensity was matched at different portion of spectral. Here, red markers represent the pesticide spectral and three different agricultural soils spectral were marked as bule, green and purple respectively. In this study, from fig. 3.1(a) the matched portion intensity was at 1050 cm-1, 1450 cm-1, 1650 cm-1, 1840 cm-1, 2920 cm -1 and 3350 cm-1 which was represent the carbon-nitrogen bond(C-N), Methyl group(-CH3), carbon-nitrogen double bond (C=N), carbon-oxygen double bond(C=O), carbon hydrogen bond (C-H) and nitrogen-hydrogen (N-H) bond respectively. According to the structure of carbendazim there are some functional groups like C-N, C=N, C=O, N-H, C-H etc. As, not only single soil sample but also every three samples were matched the functional group of pesticide so that it can be informed that, when farmers use carbendazim in their fields during cultivation it was mixed with soil and soil might be contaminated with this pesticide. At the same perspective, from the fig. 3.1(b) another pesticide named mancozeb and metalaxyl which trade name is ridomil gold that was used as fungal attack on crops and its FTIR spectral was represent with same soil samples. This time it was also shows that, there was some matched portion in the spectral between pesticide and the soil samples. Here, C-N, C-O, C=N, CH3, C=O and C-H bond were matched with pesticide functional group at 1050 cm-1, 1220cm-1, 1650 cm-1, 1450cm-1 ,1840 cm-1 and 2920 cm-1 respectively. In this study farmers share what kinds of crops and pesticide were used by them. For cultivation carrot they had used two types of pesticide named knowin and ridomil gold and their chemical name was carbendazim and Mancozeb+Metalaxyl. According to fig. 2 (a), 2.1(b) based on functional group matched it might be said that, used pesticide can remain into soil particle.

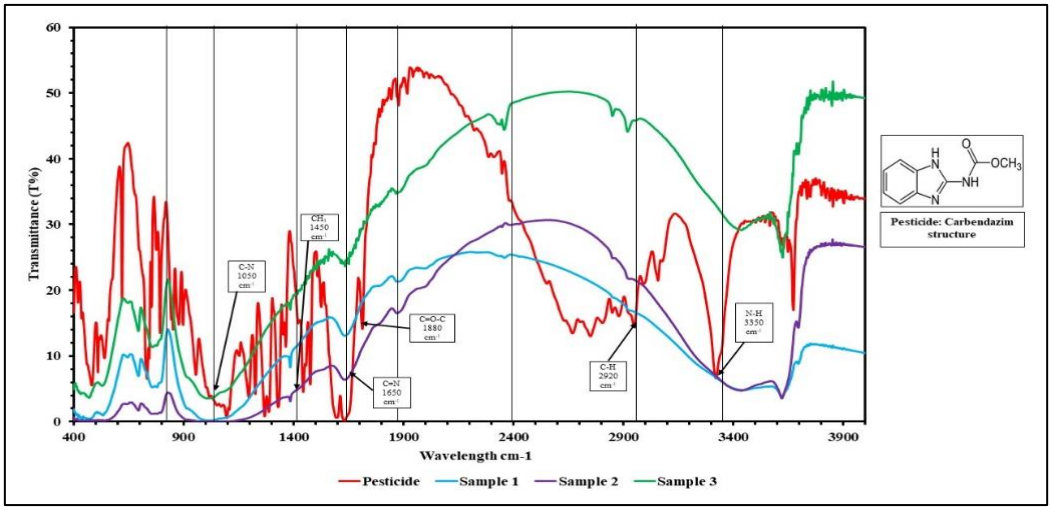

Fig 2 (a): FTIR spectra of soil and Carbendazim

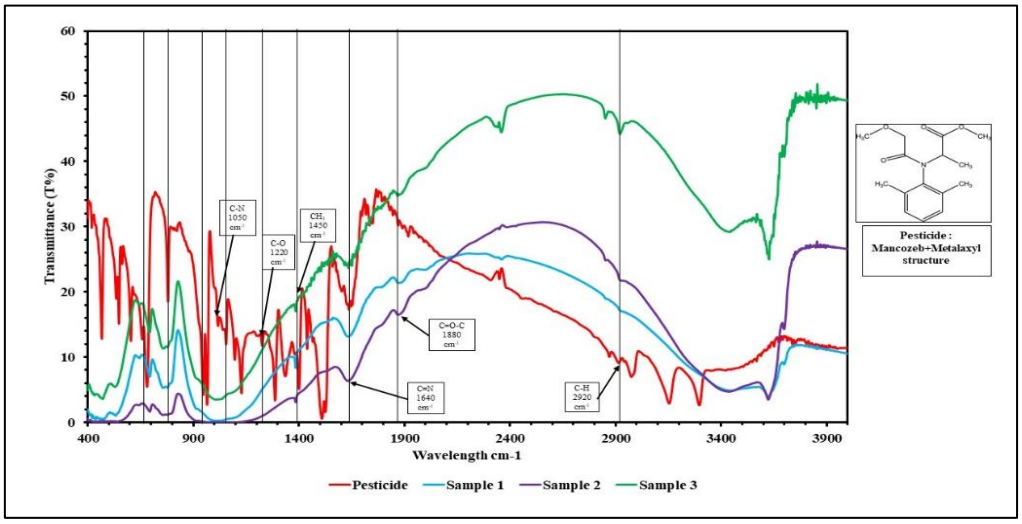

Fig 2.1 (b): FTIR spectra of soil and Mancozeb+Metalaxyl

3.2. Carrot

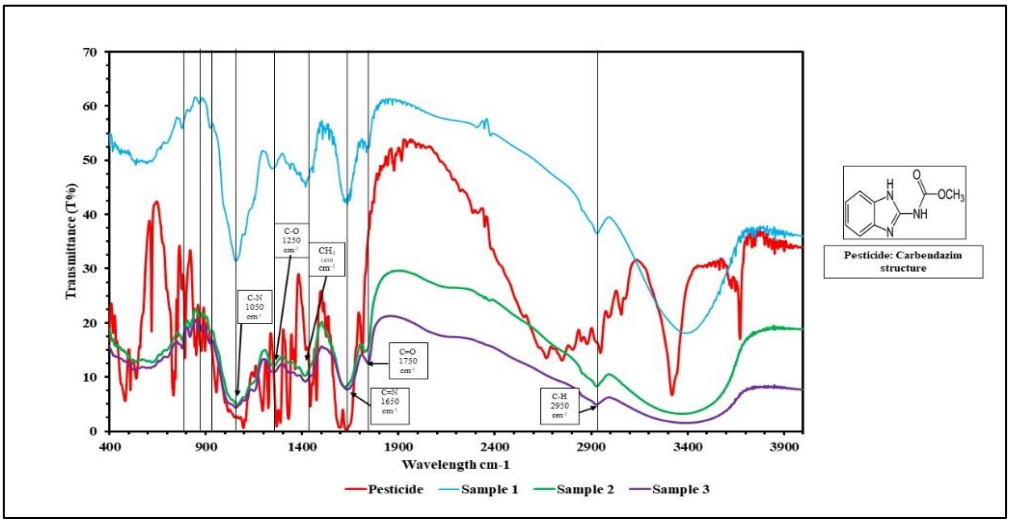

Fig 2.2 (a): FTIR spectra of carrot and Carbendazim

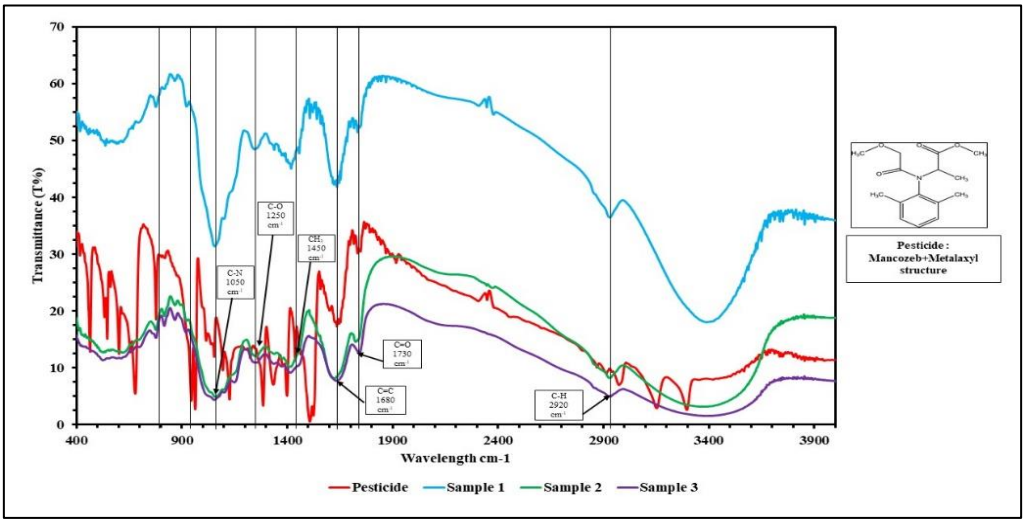

Fig 2.3 (b): FTIR spectra of carrot and Mancozeb+Metalaxyl

Figures 2.2(a) and 2.3(b) depict the findings of combining IR spectroscopy with two distinct pesticides, such as pesticides ridomil gold (Mancozeb+Metalaxyl) and knowin (Carbendazim), on a carrot indicated as a sample. These graphs all show some points in common between the sample and the pesticide, indicating that the same molecule contains both the sample and the pesticide. Vegetables may still have pesticide residue after harvest and sale since pesticides were

sprayed to them during planting. According to *(Chegg, 2003)*, the C-N bond has an IR frequency between 1020 and 1230 cm^{-1}, the C=N bond has a vibrational frequency between 1550 and 1650 cm-1, the C=O bond has a wave length between 1715 and 1745 cm, and the C-H bond has a vibrational frequency between 2700 cm-1 and 3300 cm-1 with a medium intensity. As shown in the above figure, the usual wavelength numbers are 1050 cm-1 for the C-N bond, 1650 cm-1 for the C=N bond, 1735 cm-1 for the C=O bond, and 2930 cm-1 for the C-H bond. The C-C bond, C=C bond, C-O bond, C=O bond, C-N bond, and C-H bond are all present in pesticide Mancozeb+Metalaxyl. Pure carrot does not carry a C-N bond, but after harvesting and cooking, it does. The following bonds are also present in Pesticide carbendazim: C-C, C=C, C-O, C=O, N-H, C-N, C=N, and C-H. Despite the fact that this bond is absent from the sample chemical structure, the C-N and C=N bonds are present in carrots. Since pesticide carbendazim residue is still present after harvesting, cooking, and marketing, the existence of C-N bonds and C=N indicates this. Since carrots still possess pesticide chemical bonds, this might be confirmed that vegetables acquire pesticide residue when pesticides were sprayed during the planting period.

Recommendation

The results of the study demonstrate that use of pesticide on crops might not be accurate. Firstly, in this study we found pesticides into soil sample and secondly, we found same pesticide on carrot. The surprising things that the carrot was cultivated the same soil that we examined. So, it can be said that, the residue of pesticide into soil transfer into carrot through bioaccumulation process. Another aspect is that farmers' knowledge levels are insufficient, for this reason they could not use pesticide on crops in an accurate way and this is evident in our findings. Farmers should receive ongoing instruction in pesticide safety as well as instruction on how to use personal protective equipment, maintain good personal hygiene, and keep their surroundings clean both before and after applying pesticides. Promotion of alternative pest control techniques, such as the use of bio pesticides, can also begin. Because of pesticide detrimental effects on both human health and the environment, chemical pesticides would become less necessary.

Limitation

Fourier Transform Infrared Spectroscopy (FTIR) offers a rapid and non-destructive method for detecting pesticide residues in agricultural soil and carrots. But in this study, we cover only qualitative part. How much pesticides were present on soil and carrot were not detected in this study. And first time we used this methodology for soil and vegetables. In this study only detect and justify pesticide active ingredients were present in sample after harvesting.

Conclusion

Food contamination is the biggest challenge for any developing country in the world like Bangladesh. The most widely utilized agrochemicals in the research region were WHO class II pesticides. The results show that WHO class II pesticides are the most frequently used pesticides in the study area. Total 2 number of pesticide (1 Insecticide &1 Fungicide) were found into both soil and carrot samples. Food contamination is the biggest challenge for any developing country in the world like Bangladesh. It may be stated that the pesticide management techniques of the vegetable producers in the study area were inadequate. As a result, initiatives to improve farmers' pesticide understanding through increased training are critical and should be promoted by relevant stakeholders. Furthermore, authoritarian enforcement of pesticide sales restrictions should be implemented to raise farmers' ability on safety concerns. It is also advised that adequate procedures for vegetable production, such as Integrated Pest Management (IPM), which has the potential to reduce pesticide use and chemical exposure, be implemented.

Conflicts of interest: The authors declare no conflict of interest.

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