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SAMPLING FOODSTUFFS FOR MYCOTOXIN ANALYSIS

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Abstract - The obvious reason for sampling any given lot of material is to obtain a portion for determination or observation of attributes of the particular lot. It is also obvious that the sample must be representative of the lot to obtain meaningful results. Traditional means of sampling and sample preparation of agricultural crops and foodstuffs are usually not adequate for mycotoxin analyses. The main reason for this is that mycotoxin contamination is usually of a heterogenous nature and this presents problems in the preparation of a homogenous sample for analysis. Sampling plans, sampling equipment, and sample preparation will be discussed.

INTRODUCTION AND BACKGROUND

The obvious reason for sampling any given lot of material is to obtain a portion for the determination or observation of attributes of the particular lot; in the discussion here it will be chemical analysis. It is also obvious that the sample must be representative of the lot if meaningful results are to be obtained. Traditional means of sampling and sample preparation of agricultural crops and foodstuffs are usually not adequate for mycotoxin analyses. The main reason for this is that mycotoxin contamination is usually of a heterogenous nature and this presents problems in obtaining a homogenous sample for analysis. For example, it has been shown that only a few kernels in a lot may be heavily contaminated but the rest of the kernels will not be contaminated. Therefore, in order to obtain a representative lot sample for analysis, it is necessary to take a relatively large amount from a number of sites in the lot and then properly prepare this lot sample in order to produce a representative sample for analysis.

The exceptions are for foods which are readily flowable liquids, e.g., milk or beer, and foods which have been made into pastes by the processing, e.g., almond paste or smooth peanut butter or processed into powders, e.g., flour. The first class requires only a stirring whereas the latter classes require some mixing and blending to assure homogeneity.

In analyzing foodstuffs for mycotoxins we are interested in finding out the true value (average or mean) of the contamination in any given lot. We will see later that the true value of a lot is dependent upon at least three distinct but interdependent parts; namely, (1) sampling, (2) sample preparation, and (3) analysis. The analysis part of this problem will usually have the least error associated with it; the sampling part will have the largest and the sample preparation part will usually have an error lying somewhere between these two. I will not be discussing methods of analysis except as they inter-relate with sampling and sample preparation.

Although we speak about plans for mycotoxin analysis, the plans developed to date have been devised for aflatoxin contaminations. There is little if any evidence to indicate that the nature of contaminations by other mycotoxins will be different from that of aflatoxin. So the information presented here for aflatoxin can be considered to apply to other mycotoxins until evidence is generated to show that a specific mycotoxin should be treated differently.

HETEROGENOUS CONTAMINATION

Shortly after it was recognized that the aflatoxin problem (the causative factor in Turkey x disease) was associated with peanut meal (1) the heterogenous nature of contamination of individual peanut kernels was observed; thus alerting investigators to the sampling and sample preparation problem in determining the extent of contamination of any particular lot of peanuts. The initial work (2) was done by selecting visually suspected nuts from two lots of peanuts. Aflatoxin was found in about half the selected nuts from these lots and the amount ranged from a detectable trace to a high of 1,100 μg/g with an average of 112 μg/g.

From these findings it was estimated that one highly contaminated kernel in 10,000 kernels could result in an average of 50 μg/kg of peanuts (3). Analysis of two separate 2 kg subsamples from one of the lots of peanuts revealed 30 and 400 μg aflatoxin B_1/kg respectively. Of 100 individual Brazil nut kernels analyzed, five were contaminated at levels of 0.05 to 25 μg/g (4). In an investigation of 771 cottonseed kernels (5), 18% were found to be contaminated at a range of 150-5.75 million μg/kg. Estimations made from analyses of walnuts indicated a contamination of 1 in 28,500 walnuts (6) and 1 in 29,000 hazelnuts (7).

Analyses of 256 randomly selected kernels from a known contaminated lot of corn were all negative for aflatoxin; however, 5 out of 10 selected defective kernels from the same lot were found to be positive for aflatoxin (8). In another study (9), corn kernels that showed bright fluorescence under ultraviolet light were analyzed. Most of the fluorescing kernels contained aflatoxin and some at extremely high levels, e.g., more than 400,000 μg/kg of aflatoxin. These samples clearly illustrate the point that only a few individual kernels or seeds from a lot may be contaminated and the levels of contamination may be extremely high.

The analysis of subsamples from contaminated lots illustrate the heterogenous nature of the contamination. Samples from two bins of corn ranged from 0 to 376 μg/kg for 72 samples with a mean toxin level of 21 from one bin and 0 to 332 μg/kg for 72 samples with a mean of 15 for the second bin (10). In the case for peanuts (11), analysis of ten 4.5 kg samples from a well blended lot of peanuts gave values of 0, 0, 3, 4, 4, 5, 15, 60, 160, and 165 μg/kg. The average was 36 μg/kg. Dickens goes on to vividly demonstrate the extent of the problem by displaying a clear plastic bag of rice containing 0.1% of red colored kernels and removes approximately 250 kernels by scooping a small beaker full and pouring the kernels out on a surface where the red colored kernels can be readily counted. By removing several beakers full and observing them he did not get any red kernels. He points out that from a poison distribution for this type of sampling one predicts that 78 out of 100 times you would get zero. This clearly illustrates the futility of analyzing samples which are not representative of the lot because one obtains erroneous results.

"Pockets" of contamination will usually be associated with the bulk storage of grains, oilseeds, oilseed cakes, flours or ground mixed feeds. It has been shown (12) that a major source of contamination in peanuts was brought about because of warehousing in buildings with inadequate ventilation. These problems develop particularly when peanuts are placed in storage before they are completely dry and the resultant moist air caused condensation on the roofs and sides of the buildings. This condensation then drips on peanuts forming wet spots setting up ideal conditions for mold growth when the temperature becomes right for growth. These wet spots created pockets of highly contaminated material. Wet spots can also occur from rains if there are leaks in the roof or side walls. If these pockets are not taken into consideration in sampling, erroneous results will be obtained for the aflatoxin level for the lot. These wet spot problems can be minimized by installing adequate ventilation and correcting leaks in the storage buildings.

"Pockets" of contamination may be caused by other factors such as contaminations from certain areas of a field at the time of harvest; the storage of small loads of contaminated material in with good loads in bins or silos; or wet spots in storage containers of cereal flours or mixed feeds.

These investigations with individual kernels, subsamples from lots, the demonstration with rice and the possibilities for the development of pockets of contamination all show the necessity for a sound statistical basis for the development of acceptable sampling plans.

ASSOCIATED ERRORS

Most of the published statistically designed plans have come from the efforts of the group from North Carolina State University and their colleagues (13,14,15,16). They choose to use the negative binomial distribution because it was found that the distribution of aflatoxin contaminated peanuts resembled the distribution of the incidence of contagious diseases (17) which have been described by this statistic. They use the Monte Carlo procedures (18) for determining the acceptance probabilities for aflatoxin testing programs because it is a means for accounting for interrelated factors such as multiple samples, subsamples and analyses. Their approach in attacking this problem is described in an excellent manner (19) in a publication which breaks down the total error involved with the analysis of peanuts into its component parts and expresses these specific errors as coefficients of variation (C.V.). With peanuts, the total error is the error associated with sampling, the error associated with subsampling and the error associated with analysis. These errors are dependent to different extents upon the level of aflatoxin contamination of the lot. The analytical error is the least dependent and the subsampling error is slightly more dependent but there is a great deal of dependency for sampling, particularly at levels of contamination less than 40 μg/kg. For a lot contaminated at a level of 25 μg/kg, the approximate C.V.s for analysis, subsampling and sampling are approximately 23, 35 and 110 respectively.

A similar study was conducted for cottonseed using 10 lb. (4.54 kg) samples (20). The approximate C.V.s for a lot with a 20 μg/kg level of contamination was 8% for analysis, 18% for subsampling and 100% for sampling.

In another study (21) at a 20 μg/kg level of contamination with peanuts using a 48 lb. (21.8 kg) sample, 1100 g subsample and analysis of 2 aliquots, the coefficients of variation were 60%, 18% and 16% respectively with a total error estimated to be 80%.

In a study with corn (22), the total error was broken down into four components which are: sample error, coarse subsample, fine subsample, and analysis. Using a 10 lb. (4.54 kg) sample, the coefficient of variation associated with the 4.54 kg sample, 1 kg coarse subsample (passes a #14 mesh screen), a 50 g fine subsample (passes a #20 mesh screen), and one analysis were found to be 20, 7, 0, and 28% respectively for a lot contaminated at a level of 20 μg/kg total aflatoxin. This study shows that corn is quite different from peanuts and cottonseed in that the analytical error was the greatest and the error associated with sampling was much less than the others and even less than the analytical error.

These studies draw attention to the fact that the sampling error is usually the largest contributor to the total error. So sampling is the part in which the most improvement can be made to obtain an overall improvement in the analytical results. To date this segment of the problem has received little attention compared to the analytical; this is exemplified by the fact that there are nearly 500 references for aflatoxin methods in the literature whereas there are about 25 for sampling for aflatoxins.

SAMPLING

Because of the heterogenous nature of aflatoxin contamination of most foodstuffs, choices for improvement in obtaining a representative lot sample are (1) the collection of larger representative samples, (2) the analysis of multiple samples vs. a single sample, and (3) the lowering of the acceptance level of contamination for a lot. Each of these and combinations of these three choices have been used to reduce the error and thus produce more reliable data for decision making.

Size of sample

Since the beginning of the aflatoxin problem it has generally been the practice to use considerably larger samples than usually required for food or agricultural crop analysis. It is usual to require at least 1 kg sample, and from the onset of the problem the United States Food and Drug Administration has advocated a minimum of a 15 lb. sample. One of the important considerations is the cost of the material going into the sample. This aspect will be discussed later in this presentation. The size of the lot under investigation is another factor influencing the size of the sample; in general, the larger the lot the larger the sample should be. However, as the lots become large, the size is not as important, e.g., the same size sample representative of a 40,000 lb. lot of raw shelled peanuts is representative of a 100,000 lb. lot. Over the years, the size of the sample for the control of peanuts in the United States has risen from 12 lbs., to 24 lbs., to 48 lbs., to the current three 48 lb. samples. This increase in size evolved as the need for more reliable values were required by the manufacturer. A West Germany plan requires a 5 kg sample for roast peanuts (23).

Another factor in obtaining a representative lot sample in addition to considering the relation of the nature of the contamination is the size of the individual kernels or grains. In general, a larger sample will be required for something like Brazil nuts which will weigh 8-10 g each than for peanuts which may weigh less than 0.5 g each.

Number of samples

Initially a single sample was generally used for aflatoxin control work. More recently sampling plans require multiple samples, e.g., the Swiss plan (24) calls for 10-250 gm samples of almonds and the current United States peanut program requires three 48 lb. samples (25). There appears to be advantages to the use of multiple samples, particularly when they are used in sequential plans such as the United States peanut plan (25) which will be discussed in more detail later in this presentation.

Lowering acceptance level

Lowering the acceptance level is undoubtedly an effective means of giving added assurance that a particular lot does not have a high level of contamination. However, it does not mean that the lot contains less than the acceptance level. The main disadvantage to this approach is that it reduces the number of acceptable lots offered by producers and if carried to extreme would essentially eliminate a particular food from the market place.

Sampling equipment

A general discussion of sampling and sample preparation equipment can be found in reference (26). A representative sample can best be obtained by automatic continuous samplers in situations where such equipment can be used, such as manufacturing process streams of materials. When this is not possible, e.g., when a bulk lot is in a bin, truck, box car or similar container, probe samples should be taken by means of probes which can reach to the bottom of the container. Both hand operated and mechanical probes are available for this purpose. When the lot is bagged, samples are best taken from the bags while they are being filled or emptied into containers; these samples can be scooped or hand fulls, "grabs" placed into a collection container. After the bags are closed and/or placed on pallets the job becomes more difficult, but samples can be removed by means of small triers (probes). For lots comprising a relatively small number of bags it is best to sample each bag. As the number of bags in a lot become large, a good practice is to remove material from one-fourth of the bags.

Usually the amount of sample material removed from the lot is more than required, so it is necessary to thoroughly mix this material before removing the required amount of sample.

After mixing, the material can be reduced to the required size by use of mechanical dividers or by applying the "quartering" technique.

A procedure has been worked out for sampling pistachio nuts employing only simple shop built equipment (27).

Sample preparation and equipment

Now that a representative lot sample has been obtained, the next step in the process is to produce a representative sample of this material for analysis. In general, this will involve mixing and blending of the material, coarse grinding to reduce the particle size so the material will pass a standard #14 mesh screen, mixing to obtain uniformity and subdividing to obtain a portion for further grinding to produce a flowable material which can be subdivided down to the analytical sample (25-100 g).

An important factor in studying sample preparation procedures is the problem of evaluation so that valid comparisons between different procedures can be made. The reason for this is the inherent error in the TLC analytical methods where coefficients of variation range from 10 to 35% even when run under ideal conditions. One laboratory got around this obstacle by employing radioactive kernels of the foodstuff being investigated (3,28). One or several kernels were made radioactive by placing them in a neutron activator. These radioactive kernels were then added back to the sample being prepared. After preparation, a number of analytical size samples were removed and the radioactivity of the samples measured and compared for uniformity. Two practical procedures evolved from these investigations which are presently extensively used. One employing a commercial piece of equipment known as an HVCM (Hobart Vertical Cutter Mixer) is capable of simultaneously grinding and blending a 25 pound sample of in-the-shell Brazil nuts to produce an analytical size sample with a coefficient of variation of 3% in only 2-3 minutes. It was found that the hard shell was necessary as a grinding aid to produce this degree of homogeneity. In grinding nut meats an equal portion (weight) of crushed oyster shell was added to the nut meats as a substitute for hard shell and found to be equally effective as Brazil nut shells. This procedure is used in the United States to prepare samples of each lot of imported Brazil nuts and pistachio nuts. When it is necessary to employ two or more charges from a lot sample, it is important that a weighed portion from each charge be composited to make up the sample for analyses because no practical means has been found to mix two or more charges adequately to produce a homogenous mass before removing the sample.

The other procedure to come out of these investigations employs the principle of producing a readily flowable slurry by adding a liquid such as heptane to ground kernels which is then blended and finely ground by use of a Polytron. This procedure was found to produce the highest degree of homogeneity, a 1% C.V., and can be used with samples from 500 g to 20 or more kg. It has not been employed to any extent for practical control work, however, because other less cumbersome and less costly procedures are available. The principle was employed (28) to develop a procedure using dry grinding of the lot sample, mixing and subdividing to produce a 300 g sample which is mixed with heptane to produce a slurry which is then finely ground in a Waring blender. This is the method recommended for small samples of nut meats. This slurry grinding technique using water in place of heptane was recently employed for developing a procedure for the preparation of cottonseed, peanuts, peanut butter, peanut meal, cottonseed meal, copra and corn for analysis (29).

Another significant investigation led to the development of the Dickens-Satterwhite subsampling mill for peanut kernels (30). This is a simple compact mill developed to simultaneously comminute and subsample peanut kernels at a rate of about 3 kg per minute. As the peanuts are ground through the mill, a 5% portion is continuously removed to give a representative sampling of the material passing through the mill. The only problem with this mill when used for raw peanut kernels is that the subsample produced is usually considerably larger than required for the analytical sample and the particle size is so large that when subdivided to produce the analytical sample, an unacceptably large error is introduced. This error can be eliminated by extracting the entire subsample and then taking aliquots of the extract for the usual analysis (25). This procedure is extensively used in the United States for the control of all peanuts going into food manufacture. It is estimated that nearly 100,000 lots of peanuts are analyzed in this manner each year.

Another means of dealing with the non-homogenous nature of the subsample is to process as a slurry in a Waring blender (28) as described earlier. Since the Dickens mill has been in use it has found application for a number of agricultural commodities in addition to raw peanut kernels. In some instances it is necessary to modify the screen size in order to produce the desired subsample.

Lot samples of cottonseed can be dehulled by passing them through a Bauer disc mill with the discs set wide enough to just crack the hulls of the seed (31). The seed is then passed over a small beater to separate the hulls from the kernels. The kernels are then ground by passing them through a Dickens subsampling mill (30).

Other pieces of equipment such as hammer mills, grinders, food choppers, twin shell blenders, planetary mixers, etc. have been used in sample preparation, but the HVCM and the Dickens mill are the most extensively used, particularly in the preparation of in-the-shell nuts or when the preparation of large samples are required. Both of these machines, when properly used, do the job cheaply, rapidly and efficiently.

SAMPLING PLANS

Raw shelled peanuts

The aflatoxin testing program currently in use in the United States to test all lots of raw shelled peanuts before going to food manufactures for processing is a multi-sample sequential testing plan as described by Whitaker (21). A 144 pound sample is randomly taken by continuous automatic samplers or collections are made from every fourth bag or collections are made by other approved methods from bulk containers. This lot sample is divided into three 48 pound samples. One sample is passed through the Dickens subsampling mill (30) and the entire 1100 g subsample is extracted in 3 liters of methanol-water (55:45) and 1 liter of hexane. Duplicate 50 ml aliquots of the extract are analyzed by the AOAC Official First Action Method II (26). The results are averaged and if the mean is less than 16 μg/kg total aflatoxin, the lot is accepted. If the average is greater than 75 μg/kg, the lot is rejected. If the average is between 16 and 75, the second 48 pound sample is analyzed in the same manner as with the first sample. The values from both are averaged and if the mean is 22 μg/kg or less, the lot is accepted; if the mean is 38 μg/kg or more, the lot is rejected. If the value is between 22 and 38, the third sample is processed and analyzed as the first two. This time the six values are averaged and if the average is 25 μg/kg or less, the lot is accepted and if it is more than 25, the lot is rejected. The two most important features of this testing program is that it takes advantage of the increased reliability obtained by use of increasingly large samples and in addition takes advantage of increased reliability because of multiple analyses.

Brazil nuts

In the United States each import shipment (lot) of in-the-shell Brazil nuts are sampled and analyzed before entry is allowed. The shipments are all in bags and lots range from 500 to 2,000 bags. The bags are randomly sampled on the docks or warehouses by means of a trier (probe) to obtain lot samples of 30 to 60 pounds. The entire lot sample is ground in an HVCM (3) to give a homogenous sample for analysis. Two or more charges to the HVCM are required for the larger samples; when this is necessary, appropriate weighed portions are removed from each charge to make up the required analytical sample.

The importer has the option of analyzing a "kernel only" sample. In these instances the entire sample is shelled, the nut meats are mixed with an equal weight of crushed oyster shell before grinding (the importer pays the cost of shelling).

Pistachio nuts

In the United States each import shipment of pistachio nuts are sampled and analyzed before entry into the country is allowed. The shipments (lots) usually range in size from 300 to 500 bags (70 kg each). The bags are randomly sampled by means of a trier to give the required lot sample, 30 to 60 pounds, depending on the size of the lot. The entire lot sample is then ground in an HVCM taking the same precautions as taken with Brazil nuts.

Milk and milk products

As mentioned earlier in this discussion, liquids such as beer and milk require no special sample preparation because the bulk lot and the lot sample can be readily made homogenous by simple stirring before removing the sample for analysis. Because some milk products such as yogurt, ice cream, butter or cottage (fresh) cheese are homogenous, a sample taken from any portion of the lot will give a representative sample for analysis. This principle is not valid for all cheeses however. Kiermeier (32) investigated fermented and aged cheeses and found B and G aflatoxins in addition to the M_1 which is usually the only aflatoxin found in milk as a metabolite of B_1 ingested by the cow. The B and G aflatoxins apparently are formed by fungi growing on the cheese and he found that the amounts were quite different in portions taken from different parts of the cheese. He also points out that one cheese is not necessarily representative of cheeses from a given lot so these facts should be taken into consideration in sampling cheese for aflatoxin analysis.

ECONOMIC ASPECTS

The main economic factors involved with the overall cost of an aflatoxin testing program are the cost of the material making up the sample, cost for sample preparation, the cost for the actual analysis, and in some instances the cost of shipment of samples from the point of sampling to the place that the analysis is performed. Each of these can amount to a sizeable cost and options are available to the decision-maker.

It is readily apparent that large samples are necessary to obtain reliable analytical results. The foodstuffs of concern are generally rather expensive, particularly the tree nuts, so at today's prices the cost of a sample can easily amount to $100 to $200. In addition, at these times of limited food and feed supplies in some countries, it is not wise or attractive to use these materials unwisely.

Procedures for sample preparation all change the form of the lot sample and in some instances the procedure is destructive, e.g., grinding shell and kernel in the case of hard shell nuts and the addition of oyster shell to nut meats in the use of HVCM.

The changing of form (kernels or seeds to a ground material) reduces the value of nuts in at least most instances, e.g., pecan halves are more valuable than chopped pecans and pecan flour is of considerably less value. As pointed out earlier, the Dickens mill is used extensively in the control of peanuts in the United States. The currently used sequential plan uses only as many of the three 48 lb. samples as necessary to reach a decision. One of the distinct advantages of the Dickens' mill is that only 5% of the sample is removed for analysis and the remaining ground material still has a commercial value although it is less than that of the whole kernels.

Ayres (33) suggests a plan to minimize the sample costs for pecan processing. The market place requires some small size pieces of nuts and during the chopping process to produce these, some comparatively very low value pecan meal is formed. By selecting the halves and the large pieces on a statistical basis for chopping, the resultant low value pecan meal can be used as a representative sample for analysis. Similar studies of manufacturing processes for foodstuffs with the view of minimizing sample costs are needed.

The cost of analysis must be considered in multiple sample plans in which up to ten samples are analyzed to evaluate a lot. The costs may be justified for high cost foodstuffs when chemical reagents are cheap and analyst time is not too costly.

In some instances Brazil nut samples are shipped two to three thousand miles from the point of sampling to the analyzing laboratory. Some find it economic to shell the nuts before shipping, thus saving these shipping costs.

CONCLUSION

It can be seen from the information presented that complex procedures are required to produce an analytical sample which is representative of any given lot. In several instances statistically sound sampling plans have been devised for some foodstuffs but such plans are still needed for others. The costs of sampling for mycotoxins are relatively

large compared to those for sampling for more conventional food analyses. It is highly desirable that investigations be conducted to devise economically feasible sampling plans so that suspect foodstuffs can be reliably analyzed with a minimum of loss of foods for this purpose.

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