SEPARATION OF SEEDS BY FROTH FLOTATION

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SEPARATION OF SEEDS BY FROTH FLOTATION

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INTRODUCTION

In the field of agriculture there has always been a necessity to separate seeds in order to obtain seed stock suitable for sowing. Quite often harvested seeds contain a considerable number of impurities such as other varieties or weed seeds. Some can be separated and cleaned by mechanical or other means, but sometimes it is difficult to obtain clean seed by these methods without considerable loss. The separation of seeds by froth flotation as a research problem was undertaken in order to clean seed more quickly and easily and to separate some combinations that heretofore could not be satisfactorily separated.

From an engineering viewpoint, froth flotation has long been used to separate ores of precious metals from baser elements and dross. Since this process is dependent on differences in surface characteristics and since it was known that different seeds have different surface and conditioning characteristics, it seemed entirely logical that seeds could be separated by this method of froth flotation.

The literature was reviewed to see what had been done before along this line; two flotation cells were built of Lucite in the Chemical Engineering Laboratories

here at Michigan State College. Flotation tests were run and data obtained using different seeds and different frothing and conditioning agents. Several separations of seeds reported herewith have been satisfactorily accomplished.

However, there still remains a great deal of basic experimental work to be done with the different types of seeds and flotation agents. In other cases mechanical and chemical problems must be solved before the separations reported in this thesis may be used on a commercial scale.

HISTORY

Seeds and cereal grains are commonly treated by various methods in an attempt to cleanse and purify the grain by removing weak, unfertile, and foreign seeds and by removing or inactivating disease-promoting and grain-infesting agencies. Such attempts are commonly made through means that fall into one or the other of two general classifications.

In the first classification are grouped the blowers, screens, centrifugal devices, washers and other mechanical apparatus through which the grain or seed is passed in order to classify its constituent elements according to their specific gravities or weights and sizes for the separation of a clean and uniform product. Probably the most common method [12] is by screening and fanning. This method is reasonably efficient in the removal of light weight grains and foreign seeds. This procedure results in a considerable loss of the good grains if a particularly clean separation is needed. In cleaning cereal grains for seed by this method it is often necessary to remove from one-third to one-half of the total bulk. However, this method provides no means for the removal of fungus and other disease-promoting spores which escape from the ruptured pods and become scattered and attached to the surface of the grain seeds.

In the second classification falls the treating of grain or seeds with a medicated solution designed to kell or inactivate the grain-infesting agencies. This method makes no provision for eliminating the undesirable agencies from the grain, and is also objectionable in that thorough inactivation of the agencies to be suppressed may require a long time and involve an expensive installation of equipment whan any commercial quantities of grain are to be handled.

In 1931 application was made for a patent on a "Method and Means for Separating Seeds". (8) This patent describes a method of separating seeds from seeds by utilizing a difference in the wettability of their surfaces as distinguished from differences in specific gravity, size, or shape comprising treating the surfaces of seeds having a difference in surface wettability with a reagent adapted to increase the difference in surface wettability, introducing said seeds to an air liquid surface in such a wat that momentum of the seeds normal to the surface of the liquid is substantially eliminated, and floating the less wettable seeds from the more wettable seeds.

Another patent refers to a "Solution for Use in Separating Different Seeds". (11) This solution is a mixture made up of 77 parts of sodium nitrate, 3 parts of sodium phosphate, and 20 parts of commercial

glucose and is used in separating seeds by immersing the mixed seeds in the solution.

Still another patent describes a "Magnetic Separating Composition". [4] It is a paramagnetic cementitious water-adsorbent medium suitable for use in sorting seeds and is made up of powdered sponge iron, the pores of which contain an adherent mixture including calcium carbonate and iron oxide.

There are only a few sources of information concerning the separation or cleaning of seeds by froth flotation and they are all related to the work of Theodore Earle in his laboratory at Pacific Palisades, California.

There have been two patents issued on these general subjects. The first of these was for the "Classification and Selective Separation of Plant Seeds by Froth Flotation." An abstract [9] states that seeds such as those of lettuce, clover, blue grass, etc., are immersed in water, a flotative frothing reagent such as pine oil and duPont Frother B-23 is added, and the resulting mixture is treated with pulverized insoluble materials such as sulfur, calcium carbonate, or lime as a strengthening armor for froth bubbles, and is agitated in froth held flotation apparatus for the separation of the seed material into froth concentrates and nonfloating tails.

From the patent [13] it is found that this invention

relates to a process or method for the separation and classification of seeds, and more particularly to a process for the application of froth flotation for the successful separation and classification of plant and grass seeds, and has as an object to provide an improved process whereby mixed seeds of different kinds may be separated and classified according to their respective natures, and whereby seeds of a given variety may be separated and classified according to their relative fertility and potential strength.

In carrying out such a process certain problems are met which do not arise in minerals separation. Thus certain features must be borne in mind and the reagent to be employed must be selected accordingly, e.g., as to whether the seed is to be used as a food, the effect of the reagent on the sprouting or growing qualities of the seed, whether the seed is to be immediately planted, the presence or absence of the natural husk or hull covering the seed, the character of the soil or climate obtained during the growth of the seed insofar as the same may affect the seed characteristics in the application of the process, and the bulk, mass, and density of the seed under treatment and the strength of the froth bubbles necessary to effect the separation.

It is to be noted that the froth flotation employed in the improved process differs materially from "float

and sink" methods and from separatory methods utilizing surface tension of a liquid, and the froth flotation technique includes continued agitation of the liquid body and recirculation of the material unter treatment through the liquid body to elimination of the concentrates from the liquid body and into the froth bed wherewith the concentrates are removed from the flotation apparatus.

The other patent is for "Purifying and Cleansing Seeds and Cereal Grains". (10) It describes a method of treating seeds and cereal grains such as smutty wheat for the separation and removal of fungus growths, spore cells, spores, insects and their eggs, larvae, nits and infested grains by agitating the material in a froth flotation cell in the presence of a true frother in relatively small proportions.

The patent (14) itself states that experiments have established that a thoroughly cleansed and purified grain, free from foreign matter, diseased or infested grains, and disease-promoting or grain-infesting material, may be readily and inexpensively obtained through the use of properly controlled and regulated froth flotation methods, either alone or in combination with certain of the conventional steps now employed to that end. The specific steps to be employed in the cleansing and purification of given grain material will necessarily

vary with the specific nature of such material and the degree of its adulteration and infestation, but where a thoroughly cleansed and purified product is desired, economy and efficiency can best be served by including a properly regulated step of froth flotation in the process to which the material is subjected.

In the practice of the improved method, the material to be treated may be passed directly to a froth flotation cell for agitation in the presence of suitable reagents, but in some instances it will probably be found expedient to treat the material by more conventional means for a preliminary removal of foreign matter and undesirable constituents either before or after the flotation treatment.

A first and essential novel step of the improved method, where the seeds are infested with exterior fungus or other growths involves agitation of the grain material for the purpose of and to that degree necessary to accomplish a thorough rubbing and individual washing of the material grains for loosening and detachment of contaminating material from the grain surfaces. Following agitation, the material is further agitated in froth flotation apparatus in the presence of suitable flotation reagents, such as frothers or collectors, or both, for the final and complete separation and removal as a froth concentrate of the undesirable material and agencies

detached from the grain. The feed of material to the flotation apparatus may be accomplished either through the froth bed of the flotation unit or to the liquid charge of such unit below the froth line, either method of feeding for the specific seed being productive of the desired results. The amount of any one reagent required to effect a clean separation of the grain material will vary between a fraction of one per cent and forty pounds per ton of seed.

The separated froth concentrates and tails from an initial flotation operation may be subsequently routed through further froth flotation operations accomplished with the same or different reagents, or the products from the initial flotation treatment may be dried, screened, or otherwise classified, and thus made available for seeding or milling purposes, and the froth concentrates frequently being of value as a source of certain desirable by-products, of which ergot derived from certain fungus spores is an example.

There is another use of froth flotation [3] in connection with wheat that is quite important. It is used to remove the hull of the wheat berry without removing the vitamin, mineral, and protein deposits in the bran coats just under the epidermis. This eliminates the use of synthetic vitamin concentrates in enriching flour.

Rough cleaning of the wheat is done in the usual way, and then the grains are fed to a flotation cell, or rather a battery of cells in series. The slurry of wheat and water mixed in proper proportions enters the first cell and is agitated by the impeller. Then a reagent is added to form a froth which serves to collect the thin fibrous epidermous of the wheat as it breaks away from the kernels. As the slurry flows from cell to cell, the hull material is held at the top of the cells and the grains drop to the bottoms. The froth is piped away and the hulls are used as an insulating material. The grains are dried and later ground into flour, richer in vitamins than ordinary flour.

THEORY OF FROTH FLOTATION

Froth flotation is quite different from the ordinary "sink or float" type of flotation and does not depend upon specific gravity, size, or shape. It is a means of separation that has been used in the separation of ores since 1912, and today is used for the concentration of practically every mineral that is mined.

A few terms must be defined in order to better understand the theory of froth flotation. "A frother is a substance which, when added to water, enables it to form a more or less stable froth with air. A collector for any material is a substance (generally organic) which induces it to float at the air-water interface, and in the presence of a frother to form a more or less stable froth with the material floated. An activator is a substance (generally inorganic), the addition of which induces flotation in the presence of some collector that is otherwise without effect on the material. A depressant is a substance generally that prevents a collector from functioning as such for that material." (2)

There are two different theories in regard to froth flotation, the one based on adsorption and the other based on chemical reaction. Since chemical reaction seems entirely illogical in the case of seeds flotation, only the adsorption theory will be discussed

in this thesis.

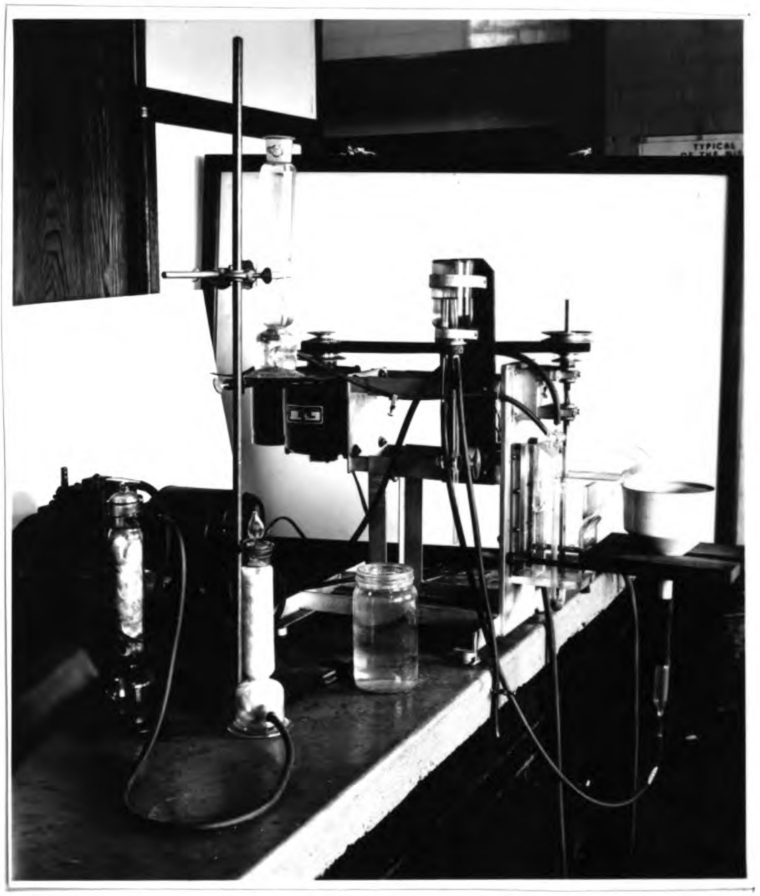
Frothing is dependent on the surface tension-concentration curves. An organic compound is usually a useful frother if, when dissolved in water, it decreases the surface tension of the water. The concentration of the frothing agent at the air liquid interface is many times greater than in the main body of the solution. Any sudden change in the size of an air bubble necessarily involves an instantaneous change in the concentration of the frothing agent and an increase of frothing power. (5)

Flotation is dependent upon oriented adsorption of the soluble collector. The adsorbed molecule cannot be considered as a whole, for only part of it is employed in holding the molecule to the surface of the material and the other part is therefore oriented away from the adsorbing surface. It is this outer part of the adsorbed molecule that determines the flotative properties of the coated material. (1) It is generally agreed that flotation is possible if one-third or more of the surface of the material to be floated adsorbs a film of the collector. The collector acts as a link between the material surfaces and air bubbles, thereby rendering flotation possible.

In the case of minerals flotation, the small

particles are covered with a film of the organic collector with the polar end of the molecule adsorbed on the surface, thereby making that surface less easily wetted by water. The non-polar end is oriented away and it is this end that allows the particle to break into the air liquid interface of the air bubble. The picture then is of an air bubble with many small particles of minerals around the inside and held to the air liquid interface by means of the adsorbed and oriented collector molecule. This forms a more or less stable froth in the presence of a frother and collects at the top of the flotation cell. Quite often one flotation agent acts as frother and collector for a given material.

In the case of seeds, probably only one or two seeds are inside a single bubble and held to the bubble wall by the collector that is adsorbed on the surface of the seed. The seeds that do not adsorb the collector remain in the liquid body in the flotation cell and are referred to as tailings or tails, and those that collect in the froth and overflow are called concentrates.



PICTURE OF APPARATUS

PROCEDURE

The research carried on was done for the most part using a flotation cell built of Lucite. Therefore the material inside the cell could be observed at all times. Two of these cells, one 100 gram and one 250 gram, were built in the laboratory of one-fourth inch Lucite sheets and fabricated by the use of a special acetone-Lucite cement. However, after a few tests were run in the smaller cell it was decided that the larger one was easier to use and could accomodate more material.

By means of an air compressor and two calcium chloride towers packed with glass wool to remove oil droplets, air was introduced through a glass tube at the bottom of the cell. The quantity of air was controlled by an outlet, fitted with a stopcock, between the pump and the cell. The cell was so constructed with baffles and a sloping outlet as to direct the froth over the edge into a Buchner funnel. The impeller was made of stainless steel and driven by a one-sixth horsepower electric motor.

These cells were equipped with a semi-automatic filtrate return. This device makes possible the re-use of the froth water and consequently reduces to a minimum

the addition of make-up water to the cell. The froth is filtered on the Buchner funnel and the filtrate accumulated in the small reservoir. The filtrate could be periodically returned to the cell to maintain the froth level. This device would be quite useful when determining reagent concentration or pH of the solution. However, during these tests, the froth level was maintained constant by the use of a water reservoir above the cell and water added constantly instead of periodically. If it were necessary to constantly add flotation reagents, it would be quite easy to do so by using this water reservoir. When the small filtrate reservoir was filled, the vacuum, created by a water aspirator, was released and the filtrate ran into the waste.

In the first tests run only one variety of seed and one flotation reagent were used. These were preliminary, and observations were made to determine which seeds would float, to what extent they would float, and with which frother or collector. After this was done, mixtures of seeds were used and actual separations accomplished with determinations of purity, loss of desired seed, and germination before and after treatment in one case.

In the preliminary tests, since the purpose was merely to observe tendencies and develop techniques,

no specific weights of seed were used and no accurate determinations were made of the amount floated or left in the cell. The quantity floated or left as tails was estimated as the nearest five per cent of the amount put into the cell.

In all the tests run, the seeds were treated before they were agitated in the flotation cell with a reagent. This treatment consisted of a thorough water wetting of all of the seed surfaces since it was very important that the seed to be separated be completely immersed at some time.

The next step was the agitation of the seeds in the flotation cell in the presence or absence of a frothing agent. Generally the seeds were added to the cell before the frothing agent, but in some cases they were added after a layer of froth had been established. In some instances better results might have been obtained by feeding the seed through the froth bed and in others by feeding the seed into the liquid below the froth bed. Even when the seed were fed through the froth bed, it was necessary to have thorough agitation in order to bring all the seed material into the liquid so that all the seeds were immersed in the water at some time.

The amount of flotation reagent was very small in most cases, so it was added by dipping the end of a wire into the reagent and then into the flotation cell.

Some of the seeds contained natural frothers, but after experimentation with this natural froth, a reagent was added in all cases.

A thickness of one-half to one and one-half inches was usually maintained for the froth bed, although in some cases it became as much as three or four inches. Only those seeds that actually came up in the froth were removed as concentrates and the balance were considered tails. In the actual quantitative determinations of the separations, the seed to be treated were accurately weighed. The seed that floated as concentrates were filtered on the Buchner funnel and partially dried by drawing air past them. The tails were removed from the cell through a hole in the bottom and filtered separately by use of the Buchner funnel. Then the concentrates and tails were dried in an oven for a short time at a temperature of fifty degrees Centigrade. When dry, the concentrate was separated into the two varieties present and the per cent of each determined. The same was done with the tails. From this data the purity and per cent loss of the cleaned seed were calculated.

DATA

DuPont Frother B-23*

	Seeds in through liquid body	Seeds in through froth bed
Alfalfa	30%	30%
Red clover	30%	30%
Corn cockle	10%	
Alsike clover	50%	
Buckhorn**	20%	20%
Catchfly	5%	
Sweet clover	40%	40%
Pepper grass	0%	
Sorrel	80%	

All percentages are the estimated amounts collected as concentrates. The seed material was agitated in the cell with the frother for five minutes.

* DuPont Frother B-23 was the flotation reagent used. All the reagents were samples furnished by the American Cyanamid Corporation.

** Buckhorn contains a natural frother that forms a very tough froth.

Pine Oil

Red clover	75% in 25 minutes
Alfalfa	30% in 25 minutes
Buckhorn	25% in 10 minutes
Catchfly	55% in 5 minutes
Dodder	1% in 5 minutes
Alsike clover	60% in 5 minutes
Corn cockle	0% in 5 minutes
Curled dock	30% in 5 minutes
Sweet clover	30% in 10 minutes
Sorrel	75% in 5 minutes
Pepper grass	0% in 5 minutes

DuPont Frother B-24

Dodder	0% in 5 minutes
Sweet clover	30% in 10 minutes
Buckhorn	10% in 5 minutes
Catchfly	95% in 15 minutes
Corn cockle	0% in 5 minutes
Alsike clover	95% in 15 minutes
Alfalfa	50% in 15 minutes
Red clover	50% in 15 minutes
Sorrel	50% in 5 minutes
Pepper grass	0% in 5 minutes
* Curled dock and red clover	75% in 5 minutes

* The tailings contain a higher per cent of red clover than the concentrates contain.

Frother 60

Red clover	90% in 15 minutes
Alfalfa	90% in 15 minutes
Alsike clover	95% in 10 minutes
Catchfly	80% in 10 minutes
Buckhorn	5% in 10 minutes
Dodder	0% in 5 minutes
Sweet clover	50% in 10 minutes
Sorrel	80% in 5 minutes
Pepper grass	10% in 5 minutes
Curled dock and red clover	95% of curled dock in 1 minute

Frother B48

Red clover	30% in 5 minutes
Alfalfa	10% in 5 minutes
Alsike clover	75% in 5 minutes
Catchfly	90% in 10 minutes
Buckhorn	5% in 5 minutes
Dodder	0% in 5 minutes
Sweet clover	50% in 5 minutes
Sorrel	50% in 5 minutes
Pepper grass	10% in 5 minutes
Curled dock and red clover	95% of curled dock in 1 minute

Frother 52

Red clover	50% in 5 minutes
Alfalfa	30% in 5 minutes
Alsike clover	80% in 5 minutes
Catchfly	80% in 5 minutes
Buckhorn	20% in 5 minutes
Dodder	10% in 5 minutes
Sweet clover	50% in 5 minutes
Sorrel	80% in 5 minutes
Pepper grass	10% in 5 minutes
Curled dock and red clover	95% of curled dock in 1 minute

Cresylic Acid

Alsike clover	85% in 5 minutes
Red clover	50% in 5 minutes
Alfalfa	50% in 5 minutes
Catchfly	80% in 5 minutes
Buckhorn	10% in 5 minutes
Dodder	5% in 5 minutes
Sweet clover	40% in 5 minutes
Sorrel	90% in 5 minutes
Pepper grass	0% in 5 minutes
Curled dock and red clover	95% of curled dock in 1 minute

Aerofloat 15

Red clover	5% in 5 minutes
Alfalfa	5% in 5 minutes
Alsike clover	10% in 5 minutes
Catchfly	20% in 5 minutes
Buckhorn	20% in 5 minutes
Dodder	5% in 5 minutes
Sweet clover	5% in 5 minutes
Sorrel	50% in 5 minutes
Pepper grass	0% in 5 minutes
Curled dock and red clover	95% of curled dock in 1 minute

Aerofloat 25

Red clover	10% in 5 minutes
Alfalfa	10% in 5 minutes
Alsike clover	50% in 5 minutes
Buckhorn	20% in 5 minutes
Catchfly	20% in 5 minutes
Dodder	5% in 5 minutes
Sorrel	80% in 5 minutes
Sweet clover	10% in 5 minutes
Curled dock and red clover	95% of curled dock in 3 minutes

Aerofloat 31

Red clover	10% in 5 minutes
Alfalfa	10% in 5 minutes
Alsike clover	50% in 5 minutes
Catchfly	50% in 5 minutes
Buckhorn	50% in 5 minutes
Dodder	0% in 5 minutes
Sweet clover	10% in 5 minutes
Sorrel	50% in 5 minutes
Curled dock and red clover	50% of curled dock in 5 minutes

In these tests the seeds were first treated with a 0.1% solution of copper acetate and washed to remove excess solution. Then while the seeds were being agitated in the cell, sodium sulfide was added. Sodium xanthate and pine oil were used as collector and frother.

Red clover	30% in 5 minutes
Alfalfa	30% in 5 minutes
Alsike clover	60% in 5 minutes
Catchfly	80% in 5 minutes
Buckhorn	10% in 5 minutes
Curled dock and red clover	30% in 5 minutes
Dodder	10% in 5 minutes
Sorrel	80% in 5 minutes
Sweet clover	20% in 5 minutes
Pepper grass	0% in 5 minutes

1. The following results were obtained by using 20 grams of red clover and 20 grams of a curled dock and red clover mixture. These seeds were agitated in the flotation cell, using Frother 60 as the flotation reagent. The concentrate was separated a second time.

Weight of concentrate	13.4 grams
Per cent red clover in concentrate	1.3
Weight of tailings	26.6 grams
Per cent red clover in tailings	94.2
Per cent loss of red clover	0.69

2* Using 20 grams of a curled dock and red clover mixture, Frother 60, and only one separation, the following results were obtained.

Weight of concentrate	14.4 grams
Per cent red clover in concentrate	24.6
Weight of tailings	5.6 grams
Per cent red clover in tailings	99.925
Per cent loss of red clover	39.0

* Germination tests were run for this separation.

3. Using curled dock and red clover and Frother 60:

Weight of concentrate	3.85 grams
Per cent red clover in concentrate	31.2
Weight of tailings	9.35 grams
Per cent red clover in tailings	99.84
Per cent loss of red clover	22.1

4. Using red clover and curled dock and Frother 60:

Weight of concentrate	6.76 grams
Per cent red clover in concentrate	47.5
Weight of tailings	31.65 grams
Per cent red clover in tailings	99.84
Per cent loss of red clover	9.25

5. Using curled dock and red clover and Frother 60:

Weight of concentrate	6.78 grams
Per cent red clover in concentrate	38.0
Weight of tailings	32.77 grams
Per cent red clover in tailings	99.85
Per cent loss of red clover	7.30

6. Using red clover and curled dock and Frother 60:

Weight of concentrate	6.16 grams
Per cent red clover in concentrate	38.2
Weight of tailings	34.00 grams
Per cent red clover in tailings	99.81
Per cent loss of red clover	6.48

7. Using red clover and curled dock and Frother 60:

Weight of concentrate	5.10 grams
Per cent red clover in concentrate	24.8
Weight of tailings	34.29 grams
Per cent red clover in tailings	99.47
Per cent loss of red clover	3.56

Tabulation of per cent red clover in tailings (purity of desired seed) and per cent loss of red clover.

	Per cent red clover in tailings	Per cent loss of red clover
1.	94.20	0.69
2.	99.925	39.0
3.	99.84	22.1
4.	99.84	9.25
5.	99.85	7.30
6.	99.81	6.48
7.	99.47	3.56

Results of the Germination Tests

At the end of one week in the germinater:

	Red clover after treatment	Red clover before treatment
Germinated seeds	173	164
Dead seeds	12	12
Hard seeds	5	21

At the end of two weeks in the germinater:

15.5% germination for the curled dock that was collected as concentrate.

18.0% germination for the curled dock that was collected as tailings.

10.5% germination for the curled dock that had not been treated in the presence of a flotation reagent.

Using wild mustard and pennycress seed, Frother 52, and agitation in the presence of the reagent for 2 minutes, the following results were obtained.

1.

Weight of concentrate	2.88 grams
Per cent mustard in concentrate	2.99
Weight of tailings	3.24 grams
Per cent mustard in tailings	90.65
Per cent loss of mustard	2.85

2.

Weight of concentrate	2.20 grams
Per cent mustard in concentrate	11.30
Weight of tailings	2.64 grams
Per cent mustard in tailings	99.36
Per cent loss of mustard	8.65

3.

Weight of concentrate	5.82 grams
Per cent mustard in concentrate	4.86
Weight of tailings	16.08 grams
Per cent mustard in tailings	88.90
Per cent loss of mustard	1.90

4.

Weight of concentrate	1.91 grams
Per cent mustard in concentrate	7.95
Weight of tailings	3.30 grams
Per cent mustard in tailings	99.27
Per cent loss of mustard	4.45

Tabulation of per cent mustard in tailings (purity of desired seed) and per cent loss of mustard.

	Per cent mustard in tailings	Per cent loss of mustard
1.	90.65	2.85
2.	99.36	8.65
3.	88.90	1.90
4.	99.37	4.45

Separation of brome grass seed from quack grass seed in order to obtain pure brome grass, using Frother 52 as the flotation reagent, gave the following results.

	Grams of brome	Number of quack seeds	Per cent of quack removed	Per cent loss of brome
1.	2	5	60.0	14.8
2.	5	5	100.0	17.9
3.	2	10	100.0	15.5
4.	2	10	60.0	9.80
5.	2	10	60.0	3.24
6.	2	10	60.0	5.18
7.	2	10	90.0	20.08
8.	2	10	80.0	41.08
9.	2	10	90.0	2.26
10.	2	10	70.0	9.88
11.	2	10	80.0	17.07
12.	2	10	40.0	2.90
13.	2	10	80.0	13.04
		Average	74.6	13.83

DISCUSSION

Some of the data obtained might be changed if the tests were to be repeated a second time. This might be especially true of some of the data obtained in the beginning of experimentation. This would be due to the fact that better techniques could be employed to obtain better results with the same seeds and the same reagents.

Some of the beginning data would be hard to duplicate within 20 or 30 per cent because there are so many variables that affect the amount of seeds floated. However, it would be possible to obtain close checks on the separations obtained if it were necessary to do so.

Factors such as thickness of froth bed, concentration of flotation reagent, size of bubbles, speed of impeller, amount of water added per unit of time, type of flotation reagent, length of time seeds are agitated in the cell before reagent is added, the amounts of seeds added, and many others, all determine the per cent of the seeds that will be floated.

According to previous work on minerals and seeds, pH is also an important factor in flotation. Tests were to be conducted to determine the extent to which pH affected the flotation of seeds; however, due to an imperfection in the Beckman pH Meter, this phase of the work was temporarily abandoned.

Better results might have been obtained in some instances by feeding the seed into the liquid below the froth bed and in some instances by feeding the seed through the froth bed. This arises from the fact that where frothers or other relatively insoluble reagents are employed, they tend to concentrate in the liquid surface immediately below the froth bed; in such a case seed introduced below the froth line will require longer agitation and the softening effect of the liquid on the seed covering may vary the normal flotative characteristics.

Undue retention of the seed in the liquid body is apt to change characteristics of the seed, and in general, if there has been a thorough immersion for a brief period, the more quickly the separation is made the better the result.

Buckhorn in particular contains a natural frother that forms a very tough froth. However, the buckhorn seeds would not float in this froth in spite of the fact that bubbles up to two inches in diameter were formed. This froth was very oily and could hardly be destroyed by pouring water over it. Several other seeds were tried with the buckhorn, but none would float in the natural froth.

With the method of adding reagent used, the concentration of reagent in the cell was always more at the beginning of the test and diminished as the froth spilled

over into the Buchner funnel. This meant that when the froth bed was first created, the air bubbles were small and they increased in size as the concentration of the frother decreased. In some instances the seeds floated better with the small bubbles and in some instances with the larger ones. Therefore, after this was determined for each variety, more seeds would be floated per unit of time if a constant optimum concentration of the reagent was maintained in the cell.

In the separation of curled dock from red clover, there is a definite optimum time the seeds should stay in the cell in the presence of the flotation reagent. In a commercial process, red clover of ninety-nine and five-tenths per cent purity is desired [15] with a minimum loss of red clover seed. Therefore, when such a purity of red clover is obtained in the cell, the process should be stopped in order to prevent further loss of red clover through floating. Experimental tests, with the cell and reagent to be used, could determine this time accurately.

For some separations it might be advantageous to recirculate the seeds through the cell two or more times in order to obtain a greater degree of purity or to decrease the per cent lost. This might require the recirculation of the concentrates in some instances and of the tailings in others. In a continuous commercial

process, the cells would be connected by pipes at their bases, or the froth allowed to flow over from one to another. In these tests the seeds were added to the flotation cell a second time for a reseparation.

In addition to a frother, oleic acid was added in one instance to act as a collector, and this seemed to increase the amount of seeds floating. Also, finely ground sulfur was added in several cases to strengthen the bubble walls and give them a protective armor, but no difference was observed in the results. Lime was added to strengthen the bubble walls and also change the pH of the solution, but no distinguishable difference was observed.

The possibility of adsorbing a copper salt on the seed, and then treating the seed as the copper salt, was investigated. The seeds were treated with a 0.1% solution of copper acetate and then washed thoroughly to remove the excess from the seeds. They were then treated with sodium sulfide to form copper sulfide that is easily floated with sodium xanthate and pine oil. This procedure resulted in no appreciable increase in the amount floated over the amount floated using pine oil alone.

The red clover germinations indicated that there was no significant difference in the per cent red clover germinated before or after agitation in the presence of

the flotation reagent. Curled dock in similar germination tests likewise indicated no interference of the flotation reagent with the life processes of the seed. The low germination of curled dock in all three cases might be due to the fact that the seeds were too old, that they were not left in the germinator long enough, or that curled dock germinations never run any higher.

In general, the per cent of red clover lost to the concentrate increased as the purity of red clover obtained was increased. However, there was no straight line function when purity was plotted against per cent lost on ordinary graph paper. This was also true in the separation of pennycress from wild mustard seed.

The usual procedure of finally drying the seed in an oven at fifty degrees Centigrade caused the natural oil in the mustard seed to be extracted. The seeds formed a hard cake on the filter paper in the oven. In order to circumvent this difficulty, these seeds were dried by placing them in a small sample bottle and blowing air over them for about ten minutes.

In the commercial field brome grass seed that contains not more than sixty-eight quack grass seeds per pound of brome grass is needed. This separation is something that cannot be satisfactorily accomplished. After further experimentation, it is believed that this

can be done by froth flotation to obtain even purer brome grass seed. In the tests performed, the results vary quite a bit, but perfect results, total removal of the quack grass present, were obtained in two cases. An average of 75% of the quack grass was removed with an average loss of only 13.6% of the brome seed. This definitely shows that a separation of the two seeds is possible.

The average of 75% removal of quack seed can be increased by several methods. First, the concentrate of brome could be recirculated through the cell and therefore leave behind more of the quack seed. Second, smaller amounts of seed material could be used in the cell and better results obtained. This is true because of the fact that the brome seed collects in one mass in the froth bed as soon as the reagent is added and, due to the nature of the seed, it is quite easy for quack seed to become inclosed in this mass. Third, a different type of cell could be used in order to prevent this mass collection of brome seed. By these means and others, 100% removal of quack grass seed from brome grass seed could be accomplished.

CONCLUSIONS

A method to separate seeds more quickly, easily, and efficiently has been worked out.

Red clover has been separated from curled dock to obtain red clover of 99.8% purity with only a 6% loss by treating the mixture in a froth flotation cell for two minutes using Frother 60 as the flotation reagent; this treatment does not affect the germination of either the red clover or the curled dock seed.

Wild mustard seed can be separated from pennycress seed to obtain mustard of 99.3% purity with a loss of only 4.5%, by froth flotation using Frother 52 and treating the seed for two minutes.

An average of 75% of the quack grass seed present in samples of brome grass seed has been removed with an average loss of brome of only 13.6%, using Frother 52. With better apparatus and better techniques 100% of the quack grass seed can be removed.

Data has been obtained that will be useful in further work on the separation of seeds by froth flotation.

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