Lab Session #4

AN Physical Properties

The main goal of this laboratory session is to provide a practical experience in the determination of the physical properties for AN and ANFO. The students will determine the desired parameters and will submit a written report within seven days from the day of the lab session (view preparation of reports section).

<table>
<thead>
<tr>
<th>Products</th>
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<tbody>
<tr>
<td>ANFO</td>
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<td>AN Prills</td>
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<td>AN Mini-Prills</td>
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1. Particle Size Distribution Analysis

This sieve analysis requires the US standard #8, #10, #12, #14, #16, #20, and pan. Start first by measuring and record individual masses of all the different sieves and pan. Next arrange the sieves, US standard #8, #10, #12, #14, #16, #20, and pan, from top to bottom. Collect 400.0g of AN prills and place them into the #08 sieve (top of stack). Cover the stack with the dust lid and the end cover. Place the entire stack into the ro-tap testing sieve shaker and turn it on for 3 minutes. Remove the sieve stack and measure the individual masses of the sieves and pans with the retained prills. Subtracting the mass of empty sieves will yield the mass that was retained.

![Figure 1. Sieve Stack with AN prills](image-url)
2. Friability Test

Place 125 numbers of 3/8 inch chrome steel spheres in a #14 sieve. Measure empty pan and put the #14 sieve with the steel spheres on top of the empty pan. Take 100.0g of the of AN prills from the retained sample in the #10 sieve from the particle distribution analysis and place it in the measured #14 sieve together with the steel spheres. Stack the rest of the sieves on top of the #14 sieve. The order is not significant for the rest of the sieves is not significant but rather to gain sufficient height so that the stack could be put in place within the ro-tap testing sieve shaker. Tape the two joints at the pan and #14 sieve to prevent mass loss before putting it into the shaker.
Turn on the ro-tap testing sieve shaker for 15 minutes. Measure the total mass of the pan and fractured prills collected. Subtract the mass from the total mass and the friability in percent mass is obtained.

**Calculations for Friability**

The friability, $f$, is expressed as mass of the fractured AN prills from shaking 100g of #10 AN prills with 125 numbers of 3/8 steel chrome balls (~3.5 g each) for 15min

$$f\% = \frac{\text{Mass of fractured AN prills}}{100g} \times 100\%$$
3. Windex Test

The objective for the WindexTM test aims to measure the fuel oil content in ANFO prills on site. It is achieved by subjecting an approximate mass of ANFO (~50g) by measuring the volume (61-64ml) using a graduated flask. Subsequently the contents are dissolved using a WindexTM, forming an immiscible mixture of FO and aqueous solution. The FO being less dense floats on the aqueous solution on this 2 phase mixture. The volume of the FO is measured by reading the top and bottom meniscus values and converted using an equation depending on the duration of stand (30min and 60min).

Sample preparation

The experiment made use of ANFO with 4% FO and 6% FO. These 4% FO content samples were synthesized by mixing 384g of AN with 16g of FO. Similarly the 6% FO content samples were produced by mixing 376g of AN with 24g of FO. The FO is slowly added to the AN and repeatedly swirl to get a good mix of ANFO. These mixture is allowed to stand for 30 minutes before testing.

Apparatus Setup and Testing Procedures

The Windex™ test as a field method only requires a graduated cylinder and a cassia flask to obtain the FO content of ANFO at site. Start first by measuring about 61ml to 64ml of ANFO sample using the graduated cylinder which correspond to an approximate mass of 50g. Subsequently transfer the ANFO into the cassia flask with the aid of a funnel making sure all of the contents get emptied. Next add Windex™ to the cassia flask to ¼ inch below the flask’s neck and slowly swirl the flask under warm water until all the prills are dissolved. The dissolving process is slow and could take up to five minutes to be completed.

It should be observed that the dissolving of the AN feels cold due to the endothermic reaction and the colour of the Windex™ becomes paler. Next add some more Windex™ to the cassia flask such that the mixture gets to the neck area. This is done to ensure that the oil phase could be measured at the graduated section. Cap the cassia flask and invert it several times to release the entrained air. Allow the mixture to stand for 30 and 60 minutes, and record the volume of FO at these two time intervals.
Calculations for Fuel Oil Content

The fuel oil content, FO content % is expressed as the following equations depending on the stand time of the mixture.

\[
FO \text{ content } \% = (FO \text{ in ml}) \times (1.748) - 1.182 \quad (30\text{min})
\]

\[
FO \text{ content } \% = (FO \text{ in ml}) \times (1.765) - 1.384 \quad (60\text{min})
\]
4. Absorption Test

The objective is to study the absorption AN prills over time, quantitatively. This was achieved through the measurement of percentage increase in mass of the AN prills that has been subjected to a controlled exposure of FO, over different times. The percentage increase in mass is defined as the absorption of the AN prills, which involves the removal the excess fuel oil on soaked prills through suction, thereby segregating the ANFO from the excess FO.

Apparatus Setup

Setup a retort stand clamping down a 250ml flask with side arm tabulation, which is attached to a vacuum pump. The 2-piece 47mm glass filters is coupled with the flask using a rubber stopper. Place a 41 ashless 70mm Whatman filer in between the 2-piece filter for ease of handling of prills, also a steel mesh is placed beneath the filter paper to hold any prill that might fall through the broken filter paper during suction.
Sample Preparation
25.0g of AN prills are placed in a 400ml glass beaker and 25ml of fuel oil (21.1g) is added to the prills. The date of time of the sample is then recorded so as to keep track of the sample’s age. The mixture will be allowed to stand at room temperature and note that the AN is fully submerged in the FO. 34 of such samples were made, however stood then tested at different ages.
Testing Procedures

Transfer all of the content into the 2-piece glass filter. Ensure all of the prills are emptied into the filter with the help of a spatula. Turn on the vacuum pump and note the time elapsed. Subject the contents to a suction of 3 minutes, after which the suction is turned off.

Measure and record the mass of the emptied beaker with a scale and tare it. Next, remove the whole 2-piece glass filter and invert it over the beaker and allowing the loose ANFO to fall within the beaker. Then, remove the clip that holds the 2-piece filter and place top portion of the 2-piece filter (holder) over the beaker. Spoon the rest of the adhered ANFO using a spatula into the beaker. Finally, inspect the filter paper and mesh for any remaining ANFO and transfer those as well. Lastly, measure and record the mass of the ANFO.
Calculations
The absorption, \( n \) is expressed as the increase in percentage of mass of the AN after absorption of FO and thereafter vacuuming the excess FO.

\[
n\% = \frac{\text{Mass of ANFO} - \text{Mass of AN}}{\text{Mass of AN}} \times 100\%
\]

Preparation of reports
The reports will be submitted as groups. It is important that a report maintains the interest of the reader while transmitting all essential information as concisely as possible. Each report should be written to suit the particular exercise under study. Do not present in the report sections that are simply a re-write from manuals or text books. Do not include any of the printed sheets from the laboratory manual in the main text of your report, unless this is a table or a question sheet that you have to fill in.

The structure of the report should include:

1. **Report Title:** Use a title page and include on this your name, group and date of experiment.
2. **Table of contents**
3. **Introduction:** This should be brief and should include a short statement of the objectives of the experiment. Basic physical principles should be outlined.
4. **Description of Apparatus:** A short description of equipment used.
5. **Procedure:** This section should only briefly outline the procedure with emphasis on the more important steps in the exercise. Draw attention to any part of the procedure, noting any difficulties encountered and how these were overcome.
6. **Results:** All measurements and derived results should be included in a suitably devised tabular form. Wherever appropriate, include a graph as this always aids in the presentation of the results.
7. **Analysis of Results:** This is the most important section of the report and hence should be given the most weight during writing. The students must predict the values using different theoretical equations. These values will be later validated using the results recorder during the lab session. Finally, the students must calculate the error between the analytical predictions and the experimental measurements, comment on the sources of error in the experiment and their effects on the results.
8. **Conclusions:** This section should contain a discussion of the results, including a critical evaluation of the experiment, the equipment used, and the techniques employed.
9. **Bibliography:** All publications referred to in the report should be listed with full details of author, title, publisher and year of publication. Clearly indicate in the text all references to these authors or reports.
Particle Size Distribution of AN Prills

**TEST PROCEDURES**

1. Measure the individual masses (empty) of US standard sieve #8, #10, #12, #14, #16, #20, and pan
2. Stack sieves in top down order from coarsest to finest with pan at the bottom
3. Prepare 400g of AN prills and dispense in the top of the stack (#08)
4. Cover the sieve stack with lid then the end cover
5. Place covered sieve stack in a ro-tap sieve shaker
6. Turn the ro-tap sieve shaker for 3 minutes
7. Remove and measure individual masses (with prills) of sieves
8. Plot percentage mass finer against the corresponding sieve size diameter
Friability Test of AN Prills

TEST PROCEDURES

1. Measure the mass of an empty pan
2. Place 125 3/8” steel spheres in a US standard #14 sieve
3. Measure 100g of the AN prills (retained in the #10 sieve from the particle distribution test)
4. Empty the 100g prills into the #14 sieve together with the steel spheres
5. Stack the rest of the sieve to gain sufficient height to be placed in the ro-tap sieve shaker
6. Ensure the pan is at the bottom with #14 immediately on top, the order of the rest of sieves are not important
7. Tape the joints at the pan and #14 to prevent mass loss as dust
8. Place stack in the ro-tap sieve shaker and start it for 15 minutes
9. Remove the stack and measure the mass of pan plus fractured prills
10. Calculate the friability base on

\[ f\% = \frac{\text{Mass of fractured AN prills}}{100g} \times 100\% \]
Windex™ Test of ANFO Prills

TEST PROCEDURES

1. Prepare 200g of ANFO in a 400ml beaker by adding a FO to AN prills. (FO should not exceed 12% by mass)
2. FO must be added slowly to AN and allow mixture to stand for at least 15 minutes
3. Measure 61-64ml (~50g) of prepared ANFO using a measuring cylinder
4. Dispense completely the measured ANFO from the measuring cylinder a cassia flask
5. Add Windex™ to fill up the bulb of the cassia flask
6. Swirl and bath the cassia flask under warm tap water to dissolve ANFO completely
7. Add more Windex™ such that it reaches the upper limit of scale on the flask’s neck
8. Stopper and invert the flask several times
9. Stand the flask to time for 30 min and 60 min
10. Measure the volume of oil phase at the 30 min and 60 min mark
11. Calculate the FO content using the following

\[
\text{FO content \%} = (\text{FO in ml}) \times (1.748) - 1.182 \quad (30 \text{ min})
\]

\[
\text{FO content \%} = (\text{FO in ml}) \times (1.765) - 1.384 \quad (60 \text{ min})
\]
Absorption Test of ANFO Prills

TEST PROCEDURES

1. Place 25.0g of AN prills in a 400ml beaker
2. Introduce 21-22ml of FO to the AN prills
3. Record the exposure time to FO of the AN prills (e.g. 1min up to 10 days)
4. Place a 41 ashless 70mm Whatman filter in between the 2-piece filter
5. Empty the AN prills and FO into the 2-piece filter
6. Apply suction for 3 minutes
7. Measure the mass of a clean and empty 400ml beaker
8. Transfer the vacuumed ANFO prills into the clean beaker
9. Measure the mass of the ANFO prills
10. Calculate the absorption using

\[
n\% = \frac{\text{Mass of ANFO} - \text{Mass of AN}}{\text{Mass of AN}} \times 100\%
\]

11. Repeat test with various exposure time