

Decomposition of Molasses Pulp Pellets in Bulk Storage

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At the Idaho Falls plant of the Utah-Idaho Sugar Company most of the molasses dried beet pulp is pelletized into $\frac{1}{4}$ inch pellets and stored in bulk in closed cylindrical tanks 85 feet in diameter by 64 feet high. The capacity of each tank is about 7500 tons of pellets. During the 1966-67 campaign, the pellets contained an average of 20.6 molasses solids and 9.0 Steffen filtrate solids percent on pellets.

In the latter part of April 1967, 3 months after the end of the campaign, the operators noticed a faint acrolein-like odor in the pellet recovery tunnel in one of the tanks. By this date the tank was about one-third unloaded. The odor was stronger in the head space above the pellets and was traced to an area about 20 feet in from the outside of the tank. Pellets were withdrawn as rapidly as possible from this area and it was soon evident that serious localized decomposition was taking place. A 16 foot square hole was cut in the tank and a large front-end loader was used to remove the pellets from the affected side. As the decomposed area was uncovered, large volumes of steam and gas evolved. The decomposition had started approximately 6 feet from the bottom and had progressed upward and outward covering an area nearly 15 feet square and about 20 feet high. The pellets at the bottom were a hard, black, glassy-looking mass. Above this, decomposition became progressively less and less until the pellets near the top were merely damp and sticky. No matter what the stage of decomposition, as soon as a fragment was broken off and exposed to the cool air, decomposition ceased. Luckily, the temperature in the affected area had not quite reached the ignition point so fire was not a problem and it was possible to make some rather detailed observations of the affected area in the tank. This led to the experimental work in the laboratory which is reported here in which the decomposition was duplicated and some of the causative factors delineated.

Equipment used in the tests consisted of two temperature controlled ovens and some wide mouth quart fruit jars with a small hole punched in the lid. Pennies were placed over these holes.

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Test No. 1

One oven was controlled at 50° C, the other at 70°. A quart bottle full of clean pellets containing no fines was placed in each oven.

Pellet moisture _____ 6.8%
Polarization _____ 15.6%

No decomposition occurred at either temperature in 10 days. On the 11th day the temperature in the 50° C oven was raised to 100° C. At the end of 4 more days, decomposition was evident at this high temperature. However, at 14 days the pellets at the 70° C temperature still showed no decomposition.

Test No. 2

In each of two bottles a fine screen was fixed in the bottles about 1 inch from the bottom. Under this screen was placed 125 ml of water. The bottles were filled with clean pellets, sealed, one placed in the 50° oven, the other in the 70° oven.

Pellet moisture _____ 6.8%
Polarization _____ 15.6%

After 4 days the pellets at 50° had taken on enough moisture to appear wet, but even after 14 days little change was detectable. On the 3rd day the pellets in the 70° oven were giving off a faint odor. This increased in intensity by the 5th day to the typical pungent acrolein-type odor. The polarization by the 10th day was 4.4 and by the 14th day was minus 5.2. The decomposition seemed to reach a constant rate at about the 6th or 7th day but the generated heat radiated out of the bottle fast enough so that the pellets did not form the charred mass noted in the tank. The temperature of the reacting pellets did not get higher than 70° C.

It was noted that decomposition started and was most complete in the storage tank where there was a heavy concentration of fines. This would form an insulating barrier to the generated heat. To simulate this condition, the next four tests were made in bottles which had been completely covered with a 3/4 inch layer of asbestos.

A sample of pellets was obtained from the area in the tank where decomposition had started. This sample contained 42% fines finer than 10 mesh. Moisture on this sample was 7.2% and polarization 12.4%.

Test No. 3

Water (125 ml) was placed under the screen on the bottom of one of the insulated bottles, which was then filled with pellets containing 42% fines. The bottle was capped and placed in the 70° C oven. On the 3rd day the temperature was 85° C and rapid decomposition was evident. By the 6th day decomposi-

tion was practically complete and the mass had the appearance and brittleness of wood charcoal. Moisture was 28% and polarization minus 5.6. If the size of the sample had been greater and the insulation more perfect, there is every indication that ignition would have taken place.

Test No. 4

Another bottle was filled with pellets containing 42% fines but this time no water was added. It was also placed in the 70° C oven. In 3 days the temperature had reached 82° C and there was a faint odor showing that the initial stage of decomposition had started. However, by the 5th day the temperature was down to 70° C where it remained for 15 days. At 15 days (the end of the test) the pellets had browned slightly and there was a faint acrolein odor. Moisture was 2.5% and sufficient decomposition had taken place to drop polarization from 12.4% to 8.7%. It appeared that the moisture in the pellets was being driven off faster than the water of decomposition was formed and this greatly slowed the reaction.

Test No. 5

A 3rd aliquot of the pellet sample containing 42% fines was placed in an insulated bottle, 125 ml of water added underneath the screen and the bottle placed in the 50° C oven. After 3 days the temperature had reached 50° C and remained constant for the 15-day test. The typical odor was noted by the 10th day which persisted at about the same level to the 15th day. By the 15th day, moisture had increased from 7.2 to 18.0%; polarization had dropped from 12.4 to 6.7%.

Test No. 6

A sample of clean pellets, free of fines but containing 15.2% moisture, was placed in an insulated bottle in the 70° C oven. No water was added. Polarization was 15.6. At 5 days the typical odor was noted. At 10 days this became quite strong and persisted to the 15th day. The temperature did not rise above the 70° C, but it was evident that slow decomposition was taking place. At the end of 15 days the polarization was 5.2% and the moisture was 8.2%.

Because moisture concentration plays such an important part in initiating the decomposition reactions, an experiment was set up to determine moisture take-up by these pellets when exposed to environments of various relative humidities. At the same time, it was desired to determine at what moisture content these pellets would mold. Saturated solutions of various chemicals to produce relative humidities from 13% to 100% were placed under bell jars along with clean pellets containing 6.8% moisture. Analyses were made each week for 7 weeks. The temperature in the

laboratory varied from 20° C to 26° C so that the humidities inside the bell jars were not exactly constant during the 49-day period. Results are shown in Table 1.

No mold appeared on any of the samples except the one at 100% relative humidity and then only when the moisture content reached 25%. On the 35th day each sample was thoroughly seeded with moldy pellets taken from the one which had molded. After 2 weeks there was no sign of further mold growth. In fact, the mold on the seeding pellets dried up.

Table 1.—Moisture uptake in pellets stored at different relative humidities—initial moisture 6.8%.

| Chemical used | Relative humidity | Percent moisture—after days | | | | | | |
|--------------------|-------------------|-----------------------------|------|------|------|--------|------|------|
| | | 7 | 14 | 21 | 28 | 35 | 42 | 49 |
| Potassium Acetate | 13 | 5.2 | 5.5 | 5.4 | 5.5 | 5.3 | 5.2 | 5.2 |
| Calcium Chloride | 32 | 5.8 | 5.6 | 5.6 | 5.4 | 5.4 | 5.5 | 5.8 |
| Zinc Nitrate | 42 | 7.0 | 8.4 | 8.4 | 8.3 | 8.0 | 8.0 | 8.0 |
| Sodium Bisulfate | 52 | 8.0 | 9.5 | 9.3 | 10.0 | 10.2 | 10.2 | 10.6 |
| Sodium Nitrite | 66 | 8.2 | 9.7 | 9.6 | 10.8 | 12.0 | 11.5 | 11.5 |
| Sodium Thiosulfate | 78 | 9.8 | 11.7 | 12.0 | 15.4 | 16.6 | 15.4 | 16.8 |
| Water | 100 | | 22.8 | 23.6 | 25.6 | (Mold) | | |

Summary

1) Pellets containing 7% moisture which are relatively free of fines show little tendency to decompose even in temperatures as high as 70° C.

2) Pellets free of fines but containing 15% moisture will decompose in temperatures as high as 70° C.

3) In the presence of 100% relative humidity, pellets of 7% moisture and free of fines show little tendency to decompose at 50° C. However, if the temperature gets as high as 70° C decomposition will take place fairly rapidly.

4) Pellets of 7% moisture but containing a high percentage of fines (30% to 40%) will decompose slowly at 70° C.

5) Pellets of 7% moisture but containing a high percentage of fines (30% to 40%) and in the presence of 100% relative humidity and 70° C will decompose very rapidly with liberation of sufficient heat to cause ignition.

6) Pellets of 7% moisture but containing a high percentage of fines and in the presence of 100% relative humidity will decompose at 50° C.

7) Pellets of the composition with which this experiment was concerned would not mold at moisture content below 25%. This leads to the conclusion that whenever pellets mold it is because water in form of condensate, rain, etc. has come in contact with them.

Conclusion

To prevent decomposition of pellets in storage, they should be produced at a moisture content of less than 10%; there should be no concentration of fines any place in the storage area; the pellets should be cooled below 50° C and held below this temperature even though supplemental ventilation through the pellet mass may be required. Pellets from production will continue to give off moisture for a time in storage. To prevent condensate dripping on the pellets, the storage facility should have enough ventilation and/or roof and wall insulation so that the walls and roof are not at the dew point for the relative humidity inside the tank. Such condensate produces mold with attendant heating, further production of water and decomposition.
