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AN EXPERIMENT IN GENERATING VINEGAR

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Respectfully Submitted as Part of Thesis Work in Mycology--
This second Minor-- for the Degree of Master of Science.

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AN EXPERIMENT IN CHEMISTRY

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An Experiment in Generating Vinegar

This experiment covers a period of three months though all that time was not actual experiment. The generator, being filled with shavings from the previous experiment, had to be emptied and cleaned. It was first thoroughly washed with water and then mopped with a solution of soda lye (1 lb. to 2 1/2 gal. of water) then rinsed clear of lye with water. The perforated head was treated in the same manner. The spiles and dump-trough were soaked in water for a day and washed with lye twice and then soaked until the generator was ready for use. The following day the generator was mopped again with lye and rinsed with water, allowed to dry and then washed with chloride of lime (1/4 lb. to a bucket of water) washed thoroughly with water and left to dry. The holes in the perforated head were carefully cleaned.

The room in which the experiment was carried on was a portion of the canning factory, partitioned off and boarded up with tongue and groove lumber, with a tightly fitting door. Two ventilating windows, 8x10 in., one in the floor and one in the side wall near the ceiling, were provided.

The generator is a large tank (4 ft. x 8 ft.) made of two inch white wood planks and heavily hooped with 5, 1/2 in. iron rods, drawn tight by lug bolts. One foot from the top of the tank is a perforated head about three inches thick, pierced by 1/8 in. gimlet holes, two inches apart to allow the vinegar stock to be evenly distributed. On this head, arranged on a pivot, is the dump

trough. It is simply a trough of two compartments and so pivoted that when one side is full it rocks over and presents the other side to the spigot. A closely fitting, movable top is arranged on top of the generator, pierced by one hole through which the vinegar stock is delivered. This cover prevents excessive evaporation and promotes general cleanliness. Six inches from the bottom of the tank is a bung-hole with downward slant and into this a siphon is fixed, made from $1/4$ in. glass tubing. This tube extended to the bottom of the generator on the inside and was covered with strips of white board to keep it from being obstructed. On the outside it also extended to the bottom of the tank. One inch above the top of this siphon ten 2 in. holes were bored, into which were driven hollow spiles which extended to within 4 in. of the center. These spiles were perforated on all sides with $1/4$ in. openings. These are to provide proper aeration of the generator. Four inches above this line of holes eight $1/2$ in. holes, with slant down, as the others, were bored and fourteen inches above this line is a third line of holes. These holes are to regulate the flow of air, without which the fermentive process can not proceed. The downward slant keeps the liquid from running out. A thermometer is inserted in one of the holes of the upper row.

Four days after the generator was washed it was put in place, about $1\ 1/2$ ft. from the floor. The spiles were sterilized by steam and put in place, the room was then tightly closed and $1/2$ pt. of sulphur was burned in it. After several hours it was opened and the siphon was put in place with strips of white wood

placed over it in such a manner as to prevent the shavings from obstructing it. After this was done about thirty five bushels of beech shavings were placed in the generator but not packed in.

These shavings are about one inch wide and are tightly coiled. This permits the liquid to come in contact with a large amount of oxygen, by which means the vinegar ferment (*Bacterium aceti*) rapidly converts the alcohol into acetic acid. The perforated head is now placed in, leveled and tightly corked around the sides with pump packing; dump trough is put in place; top put on and the generator is then ready for use.

The stock to be converted was in a room above the generator in tanks and barrels. A hole was bored through the floor and a siphon, made of 1/4 in. glass and rubber tubing, was run from the tanks, through the floor and movable cover of the generator, directly over the dump trough. The rubber tubing had a pinch-cock at the end so as to regulate the flow of stock. A nose piece was used at the end of the tubing, made by drawing out the 1/4 in. glass tube to a point with various size openings. Each size was tested so as to fill the trough at some regular interval. In this way ~~the~~ it would be self-regulating. The points would have to be very fine to do this and consequently very easily obstructed. This would be quite as much trouble as to have some one to look after it regularly hence it was d/ attended every hour by some one, putting on a certain quantity each time. In order for the self-dump to work the stock should be run through a rough filter--it would then need close attention.

Generating Vinegar

Experiment No. I

June 9th.- the first stock was run on at ~~ten~~¹⁰ am., 5 gal. of stock (bbl. No.3, 3.49% acetic acid) was run on at this time and from then until five pm., 21 gal. were run on, making a total of 26 gal. for the day. One litre of alcohol (95%) was put on with the last ten gallons. At the beginning of the experiment a thermometer was inserted in a bung-hole in the upper row of holes to register the temperature. After the experiment was well started the temperature was taken three times a day.

June 9th., 2:30 pm., temperature of the shavings 61°F. No product came off to-day--all the holes open so as to admit as much air as possible.

June 10th., 8:00 am.-temperature 63°F; 6:00 pm. it was 67°F. Bbl. No.3 was finished at noon and commenced on bbl. No. 2 (not analyzed). 27 gal. of stock was run on to-day and the shavings are not yet wet through.

June 11th., 7:30 am., temperature 69°F.-23 gal. of stock was run on to-day in which was put one litre of 95% ~~of~~ alcohol. Shavings were wet down at noon. Total number of gallons now added-76. Finished another barrel to-day and begun on the third. Cloth was placed over all the holes to-day to keep out the vinegar flies. 6:00pm, temperature 76°F.

June 12th., 8:00 am. temperature was 89°F; 10:00 am. 88°F; 4:30 pm. 87°F. 20 gal. of stock run on to-day. Some product came from the generator to-day and tested 1.7% acetic acid but it was

bad in color and flavor and it was thrown away.

June 13th., 7:30 am., temperature 95°F; 6:00 pm., temperature 100°F.

One litre of 95% alcohol added this morning. Product ran 1.9% acetic acid in am. and 2.8% in pm. Commenced to save it as the color and flavor improved.

June 14th., 6:00 am. temperature 101 1/2°F. 24 gal. of stock was run on to-day. Stopped up four of the spiles-all the other holes are still open. Product now tests 3% acetic acid and is a very good color and flavor. Finished the third barrel on 13th

June 15th. Temperature high to-day, reaching 107°F. Spiles are all now plugged with a cork pierced by 1/4 in. hole and then covered with cheese cloth. 30 gals. of stock run on to-day. Product tests 3.5% acetic acid.

June 16th.- The temperature now begins to fall; 6:00 am., 104°F; 6:00 pm., 103°F. At 10:30 am. all the bushing was removed from the spiles as it was raining and pretty cool. The product tests 2.9% acetic acid to-day. All the other holes are still stopped

June 16th., 10:00 am. the first bbl. ^{of vinegar} was filled. The barrel was thoroughly steamed before the vinegar was placed in it.

The first good vinegar came off after 96 gals. of stock had been run on the generator. After the addition of 184 gals. of stock the first barrel of product was filled. It is now running good vinegar.

Table No 1 showing daily acidity and temperatures ^{of product}

Month	Day	Temperatures			Acidity of Condensed	No of gal. run daily	
		7am	M	6pm			
June	17	102	101 1/2	101	3.36	20	
"	18	100	100	98	3.54	20	Bbl. No. 2 filled to-day
"	19	99	100	100 1/2	23.3	21	Run on 8 bbls. up to now but all not full
"	20	102	104	102	3.48	22	Bbl. No. 3 filled at 6:00 am.
"	21	100	99	99	3.70	20	
"	22	97	96	95	4.86	20	Bbl. No.4 filled at 6:00 am.
"	23	99	99 1/2	99	4.98	20	
"	24	102	102 1/2	99	4.56	18	Bbl. No. 5 filled at noon. Disc is
"	25	99 1/2	100 1/2	100 1/2	4.65	18	now stopped up and had to stop
"	26	104	103	103	6.06	20	Bbl.No.6 filled at 6am. Bels appear
"	27	101	100 1/2	100	6.06	21	There are 4 plugs out of upper row
"	28	100	100	102	5.38	18	Bbl.No.7 filled at 6 am. Lots of Bels
"	29	102	102	103	5.30	16	3 qts. of Alcohol added-all plugs on
"	30	102 1/2	101 1/2	101	5.42	20	
July	1	99 1/2	99	98	5.17		No stock run on today-all holes stopped
"	2	92	90	89 1/2	4.96		25 gal. of warm water run on today
"	3	88 1/2	88	88	3.55		30 " " " " " "
"	4	88	88	88	3.02		20 " " " " " "
"	5	87	87	86	3.76		20 " " " " " "
"	6	85 1/2	85	85	2.22		

Bbl. No. 8 filled on 2nd. and bbl. No. 9 filled on 4th. bbl. No. 70 was filled on 7th. Stopped saving vinegar at this time as there was very little acid in it. The generator has been tightly stopped ever since the first warm water was run on. Liquid is now cloudy with alk.

Eelso had now that no further attempt was made to generate vinegar. Treatment was now begun to eradicate the eels. Up to this date, 13 bbls. of stock had been run on the generator. All these barrels were not full however. From this amount of stock 10 bbls. of vinegar was taken. The best vinegar came off, in this instance, at a temperature of from 100°F to 103°F.

From July 7th. until Aug. 8th. it was a fight to exterminate the eels. At first twenty five and thirty gallons of warm water was run^{on} daily, trying to wash all the eels through but it seemed to have very little effect. It seemed impossible to get rid of them. All the holes were stopped tightly at this time. June 9th., 5 oz. of Salicylic Acid was dissolved in two buckets of water and run on the generator. This was done several times at an interval of two to three days until about 1/2 lb. was run on. This did not appear to kill the eels. The hot water treatment was kept up in the meantime. Hot water was added every day in 15 to 20 gal. quantities until Aug. 2nd. when 100 gals. of boiling water was run on in one hour. Aug. 3rd. was Sunday and no water was run on. Aug. 4th. a pipe was arranged so as to take the liquid from a bung-hole in the lower row of holes to the ventilator in the floor of the room. In this way the generator could free itself as fast as water was run on. From 10:00 am. until 12:00 am. 200 gals. of boiling water was run on the generator. From 2:00 pm. until 2:30 pm. 100 gals. more was run on. The generator was now hot through. This appeared to entirely free the generator from eels.

Experiment No. 2

The Aug. 5th.- the generator is now clear of eels and the 2nd. experiment is now begun. All the stock for this experiment was analyzed for sugar, alcohol and acetic acid. This experiment covers a period of one month. Stock contained only a trace of sugar.

Table No.2 showing alcohol and daily acidity of product, temperatures etc.

Aug.	Day	Temperatures			Acidity of stock per bbl.	% alcohol of stock per bbl.	Daily acidity of product from generator	% alcohol of this product	Approximate No. of gal of stock added daily.	
		7am	m	6pm						
"	5	110		105	2	2.45			20	Product water-not saved 2 litres of alcohol run on to-day
"	6	97	95	95	2	2.45			20	Product unfit for use
"	8	91	93	95	1.42	3.34	.30		15	No stock run on in forenoon Finished bbl No. 1 at 10 am., six of upper holes plugged
"	7	88	87	87	2	2.45			21	Finished bbl.No. 2 at 4:30 pm.Begun to save product
"	9	103	102	101	1.42	3.34	1.00		19	Pulled three plugs today-vinagar bright
"	10	97	93	96	1.67	3.38	2.22		18	First bbl.filled at 5pm 2 plugs put in at 9.30 & 1 pulled at 4:30 pm
"	12	95	94	94	.30	5.31	2.77		21	These are the vent holes in the upper row
"	13	95	96	95	.30	5.31	3.10		21	
"	14	97	96	96	.30	5.31	3.10		21	Only two vent holes open now & temperature remains at about 96°F Bbl.No 12 filled at 7am
"	15	96	95	95	.30	5.31	2.77		21	2.94% acid & .50% alcohol
"	16	95	94	94	.30	5.31	2.07	2.19	21	Vinagar becoming somewhat cloudy to-day & falling off in % of acid

Table No.2 Continued

Aug	Day	Temperatures			Acidity of stock per bbl.	Alcohol of stock per bbl.	Acidity of product daily	alcohol of product daily.	Gals. of stock added daily	
		7 am.	m.	6 pm.						
	17	93	92	90	.30	5.31	2.30	1.50	18	Bbl. No. 13 filled at noon-vinegar is a fine color today
	18	89	88	88	.30	5.31	2.55	2.18	21	Temperature falling pulled one plug-3 out now.
	19	86	85	85	.30	5.31	2.35	2.17	15	See cells today-commenced to rerun bbl. 13-pulled 1 plug.
	20	84	84	84	2.55	1.61	2.49	1.53	18	Bbl.No.14 filled at 1 pm.-4 plugs out now
	21	83	83	83	2.55	1.61	2.60	1.60	18	Put in all plugs today except two
	22	82	82	81	2.22	1.77	2.71	1.61	18	Bbl.No.13 emptied at 10 am.& commenced to rerun No.14
	23	82	82	82	2.22	1.77	2.82	1.61	18	Six plugs out now & temperature low
	24	87	88	89	2.22	1.77	3.00		18	Bbl.No.13' filled at 6 pm.
	25	89	89	90	2.53		3.50		18	Finished bbl.14 at 2 pm.& commenced running tank stock-strained it through cheese cloth .Ran on 1 litre of alcohol.
	26	88	87	86	2.53		3.99		18	pulled all the plugs from upper row & 4 from lower-13 out now.
	27	85	84	83	2.53		4.03		18	
	28	82	82	82	2.53		3.81		18	
	29	82	81	81	2.53		3.90		18	
	30	80	80	80	2.53		3.91		18	
	31	80	79	79	2.53		3.84		18	

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Experiment No. 2 continued

August 27th. there was 14 plugs out. Same date bbl. No. 14' was filled.
 Aug. 28th. commenced to run No. 13' again, making the third time it was run. Run on one half of this stock and the other half tank stock.

Aug. 29th. all the plugs out now and the temperature is still ^{low} high.

Aug. 30th. bbl. No. 13'' filled at 6:00pm

Aug. 31st. eels becoming numerous and the temperature still falling.

Table No. 2 Continued

Sept	Day	Temperatures			Acidity of stock per bbl.	Alcohol of stock per bbl.	Acidity of product daily	Alcohol of product daily	Gals. of stock added daily	
		7 am. m.	6 pm.							
"	1	79	77	79	2.53		4.05		18	
"	2	78	78	77	2.53		3.81		14	Finished bbl. 13' today & commenced on 14' bbl
"	3	77	77	78	2.53		3.78		17	No. 14'' filled at 6pm
"	4	79	80	83	2.53		3.97		18	Head stopped up today. Put in 4 plugs today - temperature rising fast.
"	5	96	99	100	2.53		4.34		18	
"	6	101	102	100	2.53		3.90		18	Eels quite numerous now - bbl. No. 15 filled at 8 am.

Table No. 3, showing the alcohol and acidity of stock after it was run through the generator three successive times.

No. of bbl.	% of acid before 1st. run	% of alcohol before 1st. run.	% of acid after 1st. run.	% of alcohol after 1st run.	% of acid after 2nd. run.	% of alcohol after 2nd. run.	% of acid after 3rd. run.	% of alcohol after 3rd. run
No. 13	.30	5.31	2.55	1.61	2.55	1.64	3.81	.37
No. 14	.30	5.31	2.22	1.77	3.34	.97		

This last table gives the results of an experiment with two barrels of good stock. This stock contained very little acid and a large amount of alcohol. It shows that stock containing that amount of alcohol and acid can not be converted into good vinegar by running it through the generator once.

From the 6th. of Sept. until the 10th. the conditions remained the same.. The experiment then ended as all the good stock had been exhausted.

On July 10th. the ten barrels from the first experiment were plugged down tight. Two double hands-full of charcoal, cracked to about the size of walnuts, were added to the barrels previous to plugging. They remained in this condition until Aug. 5th. when they were re-opened and Nos. 1, 7 & 10 still contained a good many eels. They were again plugged down with charcoal. Sept. 15th. these barrels were popped and No. 10 was free from eels Nos. 1 & 7 still contained eels. ~~At~~ Every barrel of product was plugged down with charcoal as fast as they were filled.

During the two experiments fifteen barrels of vinegar were made. In the last experiment the best vinegar was made at a temperature of from 80 - 90°F with an average of three to four plugs removed.