SOME RESULTS OF PROCESSING HEADS BY FERMENTATION [1]

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Effective utilization of the aldehydic components of the heads fraction from fruit distillery operations in laboratory-scale fermentations was reported earlier (1). The reduction or removal of the aldehydes from heads added to fermentations amounted to 75 to 95 per cent under suitable conditions. The ester component of heads was apparently not utilized by yeast although some reduction in the amount of esters did result from recycling heads through a fermentation. A general procedure for application of these findings to commercial conditions was recommended.

The effect of acetaldehyde, acetal and ethyl acetate upon alcoholic fermentation was reported in the previous issue of this journal (2). Quantities of acetaldehyde up to 0.35 per cent could be added to active fermentations without appreciable effect upon fermentation rates but, if added prior to fermentation, additions greater than 0.1 per cent resulted in appreciable time lags. Equivalent concentrations of acetal produced similar results. Both components were utilized practically 100 per cent when added to grape musts under conditions in which the fermentation of sugar was complete. A complement of alcohol approximately equivalent to the aldehyde metabolized was formed.

This report deals with some results of heads disposal under pilot-plant and commercial conditions. In addition to the results given here, verbal reports of the employment of this method for handling heads in several California wineries during the past two years have come to our attention, but without data.

PILOT SCALE EXPERIMENTS AT DAVIS IN 1955

Experiment 1. A sweet wash was obtained by adding water to unfermented pomace of Folle blanche grapes. It tested 9.1 degrees Brix and was divided into two lots of 90 gallons each. One was seeded with yeast and fermented as a control. To the other 7.2 wine gallons of heads, 190 proof, were added before fermentation.

A second sweet wash of 11.2 degrees Brix was also prepared from Folle blanche pomace. Fermentation was allowed to begin. When the Brix had decreased to 10.0, a 3-gallon sample was withdrawn as a control. Heads, prepared by diluting 16.3 wine gallons of the same lot of heads used in the first wash with water to about 20 proof, were added to the bulk of this second wash to give a final volume of 248 wine gallons.

At the end of the fermentation samples of the resulting distilling material were analyzed for alcohol, aldehydes, and esters. The data are shown in the first part of Table I. The concentrated heads* added to the first wash amounted to 7.9 per cent of the final volume and furnished two-thirds of the final alcohol content. The residual aldehyde in the heads-containing wash was less than in the control (3.8 vs. 4.1). However most of the esters

(1) Part of a report presented at the Annual Meeting of the American Society of Enologists, Asilomar, California, July 18-20, 1956.

* Concentrated here refers to the alcohol content, rather than aldehyde content. This heads contained only a little more than 0.1 per cent aldehydes while some produced in aldehyde concentrating columns may reach 10 to 15 per cent aldehydes. See cited reference 1.

contained in the added heads remained after fermentation.

The diluted heads added to the second wash furnished about 80 per cent of the final alcohol content. The aldehyde content was again lower than that of the control sample. The control, calculated to an equivalent dilution, would be lower, as indicated in the table. However, from these and other observations, the final aldehyde content of any medium of this nature seems always to amount to 1.5 to 4 mg of acetaldehyde per 100 ml, whatever may have been the amount of sugar fermented. Hence any figures calculated to other dilutions for comparative purposes are of little value.

Experiment 2. A wash of 11.0 degrees Brix was obtained from the sweet pomace of St. Emilion grapes. The fermentation was started. At the stage when the Brix had fallen to 3.8, a 3-gallon sample was withdrawn and 28.3 wine gallons (53.8 proof gallons) of the same heads used in the first experiment were added.

At the end of fermentation the volume of resulting distilling material was 315 gallons. Analytical results are shown as wash number 3 in Table I. Here the final aldehyde content was slightly higher than that of the control sample, but much reduced from the calculated amount added as heads. The fact that most of the sugars had been already fermented before the heads were added presumably accounts for the lesser effectiveness of aldehyde reduction. Curiously, however, the disappearance of volatile esters appeared to be much better in this experiment than in the former one.

Wine spirits distilled from the washes. The four lots of fermented wash produced from the above experiments were distilled into wine spirits in the 26-plate continuous column. The 12 lower plates were used for stripping and the 14 upper ones for concentrating. The product was withdrawn as a sidestream from the 24th plate (two below the top) with a simultaneous heads cut taken amounting to approximately 15 per cent of the product rate.

Analyses of the wine spirits are given in Table II. The data for the 3 heads-containing lots are lower in aldehydes but higher in esters than the other lot. This is further evidence of the efficiacy of removal of aldehyde but not of volatile esters by the addition of heads to alcoholic fermentation.

HEADS DISPOSAL AT COMMERCIAL WINERIES

At the Bear Creek Vineyard Association, Lodi, data were collected for 15 separate fermentations of distilling material containing added heads during the period October 13 to November 28, 1955. Varying quantities of heads produced in the routine operations of two distilling units were added to sweet washes, generally already in active fermentation. The volume of heads used in each trial was of the order of 1000 wine gallons. The final volume of each distilling material produced varied from 16,700 to 29,000 gallons, averaging 26,875 gallons. The aldehyde content of the heads used varied from 52 to 270 mg per 100 ml, averaging 184 and the proof range was 188 to 193, averaging 192.

Just prior to the addition of heads a sample of the sweet must was taken to the laboratory and allowed to ferment to completion. This laboratory-fermented sample was analyzed for alcohol and aldehydes. Then to this laboratory-fermented sample were added heads of the same lot and in the same proportion as used in the plant, and another analysis for aldehydes was immediately made. This should reflect the aldehyde content which the distilling material would have had without the benefit of fermentation. In comparison, the influence of fermentation upon aldehyde reduction would be reflected by the aldehyde content of the plant-fermented distilling material. The effectiveness of aldehyde reduction found by this laboratory fermentation test compared favorably with results calculated from the amount and analysis of the heads added.

The results for 3 of the trials are shown in Table III. Trial No. 6 had the highest concentration of aldehyde added as heads, No. 10 had the lowest concentration of aldehyde added and indicated the

TABLE I

Use of Heads in Wash Fermentations

Wash No.	Used Initial Brix	Treatment	Final volume of dist. mat. (gals.)	Heads (190°) added (W.gals.)	% Heads (W.gals/100 gals. DM)	Alcohol (vol. %)		Aldehydes (mg CH_3CHO/100ml)		Esters (mg EtAC/100 ml)	
						Added as heads	Found	Added as heads	Found	Added as heads	Found
1	9.1 a)	Control	88	0	0	0	4.4	0	4.1	0	4.6
	b)	Conc'd. heads added before fermentation	91	7.20	7.91	7.50	11.1	10.8	3.8	36	32
2	11.2 a)	Control	3	0	0	0	5.1 (1.81)*	0	3.5 (1.24)*	0	4.8 (1.7)*
	b)	Dilute heads added at 10.0 Brix	248	16.3	6.57	6.22	7.7	8.9	2.9	30	25
3	11.0 a)	Control	3	0	0	0	5.5	0	3.6	0	5.5
	b)	Conc. heads added at 3.8 Brix	318	28.3	8.90	8.44	13.1	12.1	4.5	40	13.1

*Values in parentheses are for the control calculated to a dilution equivalent to the dilute heads added.

TABLE III

Utilization of Heads in Commercial Distilling Material Fermentations

Trial No.	Heads added (gals/1000 gals of final DM)	Final Distilling Material					Laboratory Test				
		Ethanol (vol. %)			Aldehydes (mg/100ml)		Ethanol (vol. %)	Aldehydes (mg/100ml)		Aldehydes Eliminated %	
		Found	Added as heads	From sugar (diff.)	Found	Added as heads (calcd.)		No heads	With heads	Based on heads content	Based on lab. test
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)
6	5.89	8.2	5.6	2.6	3.5	15.9	7.7	7.6	14.5	89	90
10	1.55	7.3	1.5	5.8	1.6	2.8	5.2	2.0	3.6	42	56
15	6.85	9.3	6.5	2.8	1.5	13.7	....	1.8	14.5	89	90
Max.	6.85	9.5	6.5	5.8	3.5	15.9	7.7	7.6	14.5	89	90
Min.	1.55	4.9	1.5	1.0	1.5	2.8	3.2	1.3	3.6	42	56
Mean	4.17	7.6	4.2	3.4	2.2	7.7	5.2	3.2	9.5	67	75

TABLE II
Composition of Heads Used and Wine Spirits Produced in 26-Plate Column

Distilled Material from Wash No.*	Proof	Aldehydes	Esters	Fusel Oil
Wine Spirits		(mg per 100 ml at proof indicated)		
1 a	183	14.4	46	96
1 b	185	7.1	108	54
2 b	186	4.6	139	35
3 b	185	7.6	107	31
Heads Used	190	136	451	....

* Numbers correspond to treatment numbers in Table I.

lowest percentage of reduction while No. 15 indicated the highest percentage of aldehyde removal. Maximum, minimum, and mean values for all 15 fermentations are also reported. The percentages of alcohol from the two sources, heads and fermentation of sugar, are given in columns 4 and 5. However, the extent of the fermentation which had occurred prior to the addition of heads was not recorded. Presumably in some of the trials in which a relatively low elimination of aldehydes resulted, a major part of the alcohol had already formed before addition so there was insufficient fermentation action to reduce the aldehydes.

Columns 11 and 12 of Table III show the apparent percentages of aldehydes eliminated, calculated by two methods. These apparent percentages, while somewhat variable, can be considered very satisfactory, the mean values being 67 and 75 per cent, respectively, for the two methods of calculation. These apparent percentages were calculated without any allowance for the aldehyde content of a normal wine or distilling material. As mentioned above, it is of the order of 1.5 to 4 mg of acetaldehyde per 100 ml. If allowance could have been made for a normal content, the real utilization of added aldehydes in most of the trials would have been essentially complete. It is obvious that apparent percentages of elimination would increase in proportion to the quantity of aldehydes added.

Again from October 8 to November 9, 1956, at the Bear Creek Vineyard Association, the heads were processed by refermentation, and observations were made on 14 separate fermentation lots. A larger heads cut was taken than in the previous year, with a consequent lower average aldehyde content. Of some 16,570 wine gallons of heads returned to fermentations, representing the entire season's production, the aldehyde content varied from 71.6 to 241 mg per 100 ml averaging 126.

Table IV shows part of the results. Trial No. 1 contained the highest concentration of added aldehydes (13.9) while No. 10 contained the lowest (2.1). In both cases the aldehyde content of the final distilling material was 1.9. Of all 14 lots, only the last, as seen in the table, exhibited a final aldehyde content (7.6) which could be considered above normal. No other lot exceeded 2.9 and the average was 2.1. These values with the one exception are certainly no higher than the average aldehyde content of normal fermentations; in fact they appear to be lower. Accordingly, the removal of added aldehydes would be essentially 100 per cent if allowance were made for normal contents.

Crawford (3) reported that concentrated heads from aldehyde concentrating columns of the E. and J. Gallo Winery were processed in 1955 by adding to fermenting distilling material. By returning the heads to musts just at the start of fermentation, it was found that the elimination of aldehydes was so complete that the product of distillation was normal. Though no analytical data were available some 13,700 proof gallons of concentrated

TABLE IV.
Processing of Heads at Bear Creek Vineyard Association in 1956

Trial No.	Heads Added (Gals/1000 gals. final DM)	Distilling Material Composition				
		Ethanol (vol. %)			Aldehydes (mg/100ml)	
		Found	Added as heads	From sugar (diff.)	Found	Added as heads
1	6.14	8.9	5.6	3.3	1.9	13.9
10	3.08	4.9	2.8	2.1	1.9	2.1
14	6.93	8.3	6.7	1.6	7.6	8.8
Max.	8.38	10.5	7.1	4.1	7.6	13.9
Min.	2.21	4.8	2.1	1.4	1.2	2.1
Mean	4.79	7.3	4.5	2.8	2.1	6.1

heads were processed in this way at the Modesto plant and 11,000 proof gallons at the Fresno plant.

DISCUSSION

The best procedure for processing heads in one plant will often differ from that in another plant. Existing equipment and wine-making procedures will affect the choice. The question of the value of aldehyde concentrating columns naturally arises. As most commonly employed, aldehyde columns receive the heads cut from the primary alcohol concentrating column, and produce a relatively concentrated heads, therefore a much lesser volume. With this arrangement a comparatively large primary heads cut can be taken thereby improving the quality of the wine spirits or main product, taken as a sidestream.

Without an aldehyde column, the withdrawal of a large heads cut will require a comparatively large capacity in heads receiving tanks and, of course, the necessity of processing a comparatively large volume by recycling through fermentations. However, this comparatively large volume of heads is still so insignificant in relation to the total volume of distilling material produced that the fermentation method can be employed with but little or no outlay for new equipment.

One disadvantage in using separate aldehyde concentrating columns, aside from the investment required, is the highly corrosive character of concentrated heads. This is particularly true when the sulfur dioxide content of distilling wines is appreciable since the combination of aldehyde and bisulfite produces a highly acidic substituted sulfonic acid (1). Accordingly, the copper or other metal pickup from condensers, receiving tanks, etc. may be very large, resulting not only in early equipment failure from corrosion but possible difficulty in any fermentation disposal procedure owing to metal toxicity.

At present, we would recommend the continued use of existing aldehyde concentrating columns for moderate concentration of aldehydes followed by fermentation disposal. In plants without such columns, we recommend fermentation disposal of first run heads and believe the procedure in itself will constitute a very satisfactory answer to the heads processing problem.

The essential condition or limitations to be observed in the processing of heads by a fermentation procedure are reviewed as follows:

1. The total potential alcohol content of the medium or wash from fermentation and heads added should not exceed 10 volume per cent.

2. Heads should be added after the growth phase of yeast, i.e., after fermentation has reached a vigorous stage, but before most of the sugar has been fermented.

3. The maximum total free and combined aldehyde content, expressed as

acetaldehyde, of the wash resulting from the added heads should not exceed 0.3 per cent (0.1 if added before fermentation has begun).

4. The maximum ester content, as ethyl acetate, in the medium should not exceed about 1 per cent. It is seldom that ester content would ever be a limitation.

The presence of excessive metal concentrations, especially copper, or high sulfite in concentrated heads may require some modification of the limits mentioned.

SUMMARY

1. The process for disposal of heads by fermentation of distilling material was tested under pilot plant conditions. The reduction of aldehydes added as heads was essentially complete, when added either before or during early fermentation.

2. Data for the successful utilization of this procedure for two years in a California winery are reported.

REFERENCES CITED

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