MACERATION AND DEFECATION IN CIDER-MAKING.

I. CHANGES OCCURRING IN THE PECTIN AND NITROGEN CONTENTS OF APPLE JUICES.

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INTRODUCTION

English and French sweet ciders differ materially in character due to the difference in national tastes, to the cider apple varieties grown and to the cider-making methods employed in the two countries. In England, the juice is usually allowed to ferment to dryness and then sweetened with cane sugar before bottling, giving a light- to medium-bodied cider in which acidity, tannin and sweetness are balanced. In France, a full-bodied, low-acid, bitter-sweet cider is often preferred (1). A naturally sweet cider is considered to have the required characteristics, and great attention is paid to the pretreatment of the juice to assist in control of the subsequent fermentation. One method of fermentation control commonly practised in France (4) is defectation of the juice and this is often preceded by maceration of the freshly-milled apple pulp.

During the 1949–50 cider-making season, large-scale tests of the maceration and defecation treatments were carried out at the Research Station, using three of the four main types of cider apples grown in England,

for the purpose of assessing :-

(a) the effects of these treatments on the composition of the juice;

(b) the degree of control they afford to the subsequent fermentation;

(c) the quality of the ciders produced.

With the continuing shortage of cane sugar any method (such as defecation) offering a means of conserving sugar is of obvious importance.

As the processes of maceration and defecation have been described in detail in earlier reports by Charley (2) and summarised by Challinor (1) and Pollard (3), a very brief outline only will be given here. The term maceration has often been used to denote the mixing of either fresh apple pulp or oncepressed pomace with water, cider, or apple juice, but the authors prefer to regard such treatment as a diffusion process. The term maceration has been reserved to describe the process in which freshly-milled apple pulp is allowed to stand without any addition for a few hours before pressing (5).

To assist subsequent defecation small quantities of calcium carbonate and sodium chloride are added to the expressed juice which is maintained below 10° C. in either open or closed tanks. If the process of defecation proceeds satisfactorily a natural clarification occurs giving rise to a brilliantly clear juice between a brown jelly head and a deposit or "lees." In this paper the term defecation is used to describe this process which occurs after the addition of the salts. In previous Annual Reports, the defecation process has been described as "keeving," a treatment which is still used to some extent in England, but the two processes are not necessarily identical. No salts are added during "keeving" and the degree of clarification obtained depends very largely on the types of fruit used. Thus, if the apples contain large amounts of pectin and pectin esterase (pectase) then the juice submitted to "keeving" will, if left long enough, clarify naturally and a brown jelly head may form. Such a process is essentially a defecation without the addition of salts. If, however, the apples contain only small amounts of pectin and pectase, the juice remains cloudy and when fermentation begins a white or brown head of foam is formed in which a layer of brown apple pulp particles may often be found.

The clarified juice formed during the defecation process is carefully siphoned or pumped from between the brown jelly head and any "lees" which may have formed into a clean tank to ferment. The juice treated in this way has a reduced nitrogen content and microbial population and tends to ferment more slowly than the same juice untreated. Furthermore, fermentation often ceases before all the sugar has been attacked so that the cider remains naturally sweet.

A study of the principles governing maceration and defecation of French cider apple juices was started by the late Professor G. Warcollier (4). The work in France has been continued by Tavernier and Jacquin who have studied the effect of maceration and defecation on the nitrogen, sulphur and phosphorus contents of several apple juices (5). Charley (6) investigated the effects of "keeving" at two temperatures and showed that a better clarification was obtained at 2° C, than at 9° C. Studies of the action of the pectase naturally present in apple juice and of the associated pectin changes have been made by Kieser, Pollard and Stone (7) and Pollard and Kieser (8). This work demonstrated clearly that natural clarification could be effected by the enzymes present in the apple juice itself, irrespective of any enzymes elaborated by micro-organisms present in the juice. The initial concentration of pectin and pectase was shown to vary with the variety of apple. There was an indication also that the pectase concentration in the apple juice, as well as the pectin content, varied with the stage of maturity of the fruit. The activity of the pectase increased with increasing pH to a maximum approaching neutrality (pH 6.6). It was shown that successful defecation could occur at the pH values normally found in apple juice (pH 4.0 to 4.5) and Le Corvaisier (9) has shown that the defecation process can occur at as low a pH as 3.4. In general, however, the lower the pH the greater is the length of time needed for the onset of defecation, i.e. for a given concentration of pectase. At any particular pH the degree of pectase activity is also influenced by the concentration of the cations present (8). In this connexion Bejambes and Tavernier (10) have shown that an adequate concentration of calcium ions is necessary for efficient defecation, presumably in order that the pectic acid formed by the enzymic demethylation of pectin may be converted into an insoluble calcium derivative.

EXPERIMENTAL.

Cider-Making Procedure.

The varieties of cider apples used in the four series of experiments were as follows :-

Bulmer's Norman	Bittersweet	 milled	10.10.49
Sweet Alford	Sweet	 milled	24.10.49
Red Foxwhelp	Bittersharp	 milled	17.11.49
Kingston Black	Medium Bittersharp	 milled	28.11.49

Four treatments were given in each series, namely :-

Control (C). Juice from freshly-milled pulp, i.e. no special treatment. Defecation (D). Juice from freshly-milled pulp, to which salts were added. Maceration (M). Juice from macerated pulp.

Maceration plus Defecation (M + D). Juice from macerated pulp to which salts were added.

The casks (pipes) used were cleaned and steamed one hour before use. and kept in the cider-house.

Approximately two tons of sound, mature fruit were milled for each variety. One half of the pulp was pressed immediately and the juice, after bulking in a slate tank, was thoroughly mixed and tested for uniformity by carrying out determinations of specific gravity and titratable acidity on samples drawn at different depths in the tank. The juice was then distributed uniformly between a normal horizontal pipe (i.e. the Control C) and a vertical open-headed pipe for defecation (D). Defecating salts, 0.03% calcium carbonate and 0.04% sodium chloride (i.e. 4.8 ozs./100 gallons and 6.4 ozs./ 100 gallons respectively) were dissolved in a small quantity of the juice and added to D with thorough mixing.

Open wooden tubs 2 ft. 6 ins. deep were filled with the remaining unpressed pulp, the surface of which was then smoothed over. The pulp was allowed to macerate in the tubs for twenty-four hours and then pressed. The expressed juice was bulked and distributed as before, into a horizontal pipe (i.e. the macerated juice M) and a vertical open-headed pipe for defecation (i.e. M + D).

In order that visual observations could be made, 21-gallon clear-glass jars were filled at this time with representative samples of juice from each pipe. Each jar stood by the side of its corresponding pipe during the remainder of the experiment.

When successful defecation occurred, the brilliant juice was siphoned from under the brown jelly head into steamed horizontal casks and allowed to ferment. Unfortunately, no means were available for controlling the temperature of the juices during the defecation process.

When the specific gravity of the fermenting macerated and defecated juice of Sweet Alford and Red Foxwhelp had fallen to S.G. 1,025, samples from the fermenting juices of the four treatments of these two varieties were removed. The remainder of the slowly fermenting juice in each cask was covered with a layer of liquid paraffin (B.P. quality) to prevent development of acetic bacteria due to the large air space caused by removal of the sample. Each sample was centrifuged using an Alfa-Laval laboratory separator, and allowed to mature for one month in filled and sealed three-gallon glass jars. Each cider after maturing was filtered through Carlson K5 sheets and divided into two equal parts; one part was left unsweetened and the other sweetened with sucrose syrup to the same specific gravity as the macerated and defecated cider of that series. Samples of each were bottled for natural conditioning and the remaining unsweetened and sweetened ciders were artificially carbonated at 30° F. and 10 lbs. $\rm CO_2$ pressure per sq. in., sterile-filtered through Carlson EK. sheets, bulked and bottled. All the ciders were bottled in champagne pints, previously sterilised with 2% sulphur dioxide solution, and stored in a cool cellar for approximately six months before tasting.

Visual and Other Observations.

Maceration.

The system of maceration employed (i.e. relatively deep layers) was essentially anaerobic and according to Warcollier (4), a greater extraction of pectin and pectase occurs under such conditions without the simultaneous removal, by oxidation, of a large proportion of the tannin from the expressed juice. Aerobic maceration for the same length of time would have caused extensive oxidation and encouraged acetification. The pulp in the tubs after the period of maceration was free from obvious acetification and was apparently only slightly oxidised, i.e. browning occurred at the surface and below to a depth of one inch. The remainder appeared to be slightly lighter in colour than the original unmacerated pulp.

Table I shows analyses carried out on the fresh juices from unmacerated and macerated pulps.

TABLE I.

Effect of Maceration on the "Tannin" and Volatile Acidities of the Expressed Juice. (Results expressed as g. per 100 ml. of juice).

		Bulmer's Norman	Sweet Alford	Red Foxwhelp	Kingston Black
" Tannin "	Before Maceration (C)	0.35	0.13	0.20	0.14
	After Maceration (M)	0.32	0.12	0-18	0.10
Volatile Acidity	Before Maceration (C)	-	_	0.035	0.011
as acetic acid)	After Maceration (M)	_	_	0.032	0.013
Duration of Mace	ration	14 hours	22 hours	16 hours	27 hours

With the exception of Kingston Black in which the pulp was held in the tubs for the greater length of time, maceration had little significant effect on the tannin of the juice subsequently expressed. Volatile acidity did not alter appreciably, i.e. for either of the two varieties examined.

Defecation.

While one of the objects of defecation is to secure a clarified juice, in practice the most obvious sign of successful treatment is the formation of a

compact, brown, pectin-jelly head and it is necessary to sample the juice under the head before the degree of clarification can be assessed. Plate XIII, Fig. 1 shows a typical brown jelly head formed during successful defecation and Plate XIII, Fig. 2 shows a white head as formed by the Controls and other juices which did not defecate.

A summary of the defecation treatments that gave rise to brown jelly heads and to clarification is given in Table II together with the time taken for the heads to form. All other treatments gave white heads and the juices did not clarify.

TABLE II.

TIME TAKEN FOR DEFECATION TO OCCUR.

Variety '	Treatment	Time taken for the formation of a brown head
Bulmer's Norman	Defecation only	2 days
Dulliti o Italiana	$\begin{array}{c} \text{Maceration plus Defecation} \\ \text{(M + D)} \end{array}$	٦.,,
Sweet Alford	Defecation only (D)	3+4 "
The state of the s	Maceration plus Defecation (M + D)	2 ,,
Red Foxwhelp	Defecation only (D)	7 ,,
	Maceration plus Defecation (M + D)	3 ,,
Kingston Black	Maceration plus Defecation (SO ₂ treated) juice (M + D + SO ₂)	18 "

Notes: 1. Three days after pressing, the Sweet Alford juice to which defecating salts had been added (D), formed a white foam head above a cloudy juice. This juice was siphoned into a clean, freshly steamed, horizontal cask and four days later the juice extruded a large quantity of brown, pectin-jelly clots through the bunghole of the cask, leaving a brilliant juice.

2. With Kingston Black, defecation was very slow and only macerated and defecated juice to which 60 p.p.m. SO₂ had been added initially to restrict fermentation, eventually developed a brown head 18 days after pressing. However, the head that formed was very thin and loose and the juice underneath was not brilliant.

Analytical Methods.

The following determinations were made on samples of the fermenting juices and ciders at suitable intervals and references are given for the methods employed:—Specific Gravity (14); Titratable acidity (11); Volatile acidity (12 and 13); pH (glass electrode); "Tannin" (12); Rate of Fermentation (14); Sulphur Dioxide (13); Total Reducing Sugars (14).

Nitrogen: Determined by micro-Kjeldahl, expressed as mg.N/100 ml. (14). Soluble nitrogen was determined after centrifuging at 2,500 p.p.m. for 30 mins.

Pectin: Determined by Carré and Haynes method on centrifuged juice before and after precipitation with acetone (15). Expressed as calcium pectate g./100 ml.

RESULTS.

For each variety, the total quantity of fruit needed to provide the juice for all four treatments was milled on the same day, and half of the milled pulp was pressed immediately for the Control and Defecation treatments. The juice from the macerated pulp was not expressed until the following day, i.e. for the Maceration and Maceration plus Defecation treatments. Since the remaining treatments were carried out on the juice, the date of pressing was regarded as the start of each treatment and the time-references in text, tables and figures have been reckoned from that date.

Pectin Changes due to Maceration and Defecation.

Analyses of both crude and purified pectin were carried out on Sweet Alford and Red Foxwhelp juices. The analyses showed the same trends for all treatments although the crude pectin was always slightly greater in amount. Maceration of the pulp had no effect on this difference in either variety. All estimates of pectin quoted in the remainder of this section relate only to purified pectin.

In Table III are given the pectin contents of the juices freshly expressed from the unmacerated and macerated pulps of Sweet Alford, Red Foxwhelp and Kingston Black.

TABLE III.

Effect of Maceration on the Pectin Content of Fresh Juices.
(Results expressed as g.calcium pectate per 100 ml. juice).

Variety	Pectin	Content	Change
variety	Before Maceration	After Maceration	Pectin Content
Sweet Alford	0.047	0.072	+0.025
Red Foxwhelp	0.090	0.139	+0.049
Kingston Black	0.077	0.089	+0.012

The initial pectin content of the fresh juices differed for the three varieties. Maceration of the pulp caused a significant increase in the pectin of the expressed juice in each variety. Sweet Alford and Red Foxwhelp showed the greatest increase and in these varieties, the subsequent defecation was successful.

The pectin changes that followed the different juice treatments are shown for Sweet Alford in Table IV.

TABLE IV.

EFFECT OF DEFECATION ON THE PECTIN CONTENT OF FRESH JUICE.

SWEET ALFORD.

	pH of		Pectin Conter ectate per 100		Rate of Pectin
Treatment	Juice After Treatment	At Time of Pressing	2 Days After Pressing	7 Days After Pressing	Removal per day (as mg. Calcium pectate per 100 ml. juice)
No Treatment	4-1	0-047	0.039	0-019	4
Defecation only	4.4	0.047	0.025+	None*	11
Maceration only	4-1	0.072	0-057	0.030	7
Maceration plus Defecation (M + D)	4-4	0.072	None*	None	36

Estimation carried out on juice siphoned off from under the white head.
 Estimation carried out on clarified juices after successful defecation.

It is evident that the process of defecation effectively reduced the pectin content of the treated juices; thus, in the defecated juices D and M+D, pectin was absent, e.g. after only two days for M+D. The rates of pectin removal for the first few days after the four treatments have been calculated and are shown in the right hand column. In the juices submitted to defecation, D and M+D where ample calcium ions were added initially, the increased rate of pectin removal in the juice from macerated pulp (M+D), as compared with the juice from unmacerated pulp (D), indicated a greater extraction of pectase from the pulp during maceration. This was also suggested, but to a lesser extent, by the corresponding figures for the non-defecated juices (C) and (C) and (C) the rate of pectin removal being greater after maceration. The slower rate of pectin removal in the non-defecated juices as compared with defecated juices, may be ascribed either to a deficiency in calcium ions or to the lower pH. The successful defecation of Red Foxwhelp juice at the low pH of 3·3 would, however, suggest that the pH is not the decisive factor for this juice.

Despite an unfavourable pH (average 3-3), the rate of pectin removal in Red Foxwhelp juice was slightly greater for each of the four treatments than in Sweet Alford juice. A possible explanation of these results is that Red Foxwhelp contained a larger concentration of pectase than Sweet Alford. Other tests showed that the fruit sample of Red Foxwhelp was in fact high in pectase.

Nitrogen Changes due to Maceration and Defecation.

Samples for analysis were withdrawn from the centre of each pipe without disturbing the lees; thus, the analyses relate to the fermenting juice and the nitrogen content of the lees is not included. The following interpretation of the nitrogen data and its conversion into data representative of yeast,

is that used by Burroughs and Challinor (16). This conversion is true provided the micro-organisms present consist almost entirely of yeasts and do not include more than a small proportion of bacteria. From the few microscopical observations carried out on each of the fermentations of the Sweet Alford juice, it appeared that the material in suspension did in fact consist almost entirely of yeasts.

Thus, for the Controls in each of these experiments, i.e. the untreated juices which were allowed to ferment naturally, the total nitrogen in any sample is made up of the soluble nitrogen plus nitrogen in suspension. Nitrogen in suspension, which may be regarded as representative of yeast in suspension in any particular sample, is obtained by subtracting the soluble nitrogen from the total nitrogen of that sample. Nitrogen in the lees, regarded as representative of yeast in the lees at any time, is not determined directly but is obtained by subtracting the total nitrogen at the time of sampling from the initial total nitrogen of the juice. An indication of the amount of yeast which has grown up to any particular time is given by subtracting the soluble nitrogen at that time from the initial soluble nitrogen of the fresh juice.

Nitrogen Changes in the Untreated Juices (i.e. Controls).

These changes in the distribution of the nitrogen, regarded as representative of the changes in yeast distribution, are shown diagrammatically in Fig. 3, using data obtained from the fermentation of the Control Sweet Alford juice, since this fermentation was examined more fully in this respect than the other fermentations. All nitrogen data have been expressed as mg.N/100 ml. of juice or cider and have been abbreviated to mg. in the text.

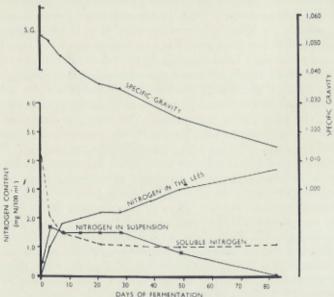


Fig. 3. Fermentation of the Control (Untreated) Sweet Alford Juice showing changes in Specific Gravity and Distribution of Nitrogen in the Fermenting Juice.

During the first eighty days of this fermentation, the specific gravity decreased from an initial S.G. 1,053 to S.G. 1,016. The rate of fermentation during the first three days was slow, a fall of two degrees in S.G., but the soluble nitrogen of the juice decreased very considerably, from 4.4 mg. to 2.1

mg., i.e. a loss of 52%, indicating an extremely rapid utilisation of soluble nitrogen for yeast growth. Soluble nitrogen decreased more slowly to 1·1 mg. from the 3rd to the 21st day, i.e. down to 25% of the initial soluble nitrogen content. Thus, during the first part of the fermentation, occupying twenty-one days, although there was a loss of only 16 degrees in specific gravity, representing one-third of the total decrease in S.G., the greatest utilisation of soluble nitrogen occurred and the remainder of the fermentation proceeded with practically no further decrease in this fraction.

Nitrogen in suspension increased rapidly to a maximum of 1.7 mg. on the 3rd day and then after a very slight fall, remained steady at 1.5 mg. for a further twenty-one days. From the 28th day onwards, there was a steady decrease in the amount of this fraction and on the 83rd day it was negligible. Nitrogen continued to accumulate in the lees throughout the eighty-three days, but the rate of accumulation for the first seven days was very much faster, approximately eight times, than it was from the 7th to the 83rd day. Thus, in a Control fermentation, loss of soluble nitrogen is representative of growth of yeast, which either remained in suspension or accumulated in the lees; this nitrogen remained in the fermentation vessel.

Nitrogen Changes Due to Maceration.

The Sweet Alford juice freshly expressed from macerated pulp had a slightly lower soluble nitrogen content than juice from unmacerated pulp; 4.0 mg. and 4.4 mg. respectively, i.e. a difference of 9%. In the juices of the other three varieties however, the soluble nitrogen increased slightly after maceration, as illustrated by the nitrogen-data for Red Foxwhelp shown in Table V.

TABLE V.

Effect of Maceration on the Nitrogen Contents of Fresh Juice.

Red Foxwhelp.

(Results expressed as mg.N/100 ml. juice).

Nitrogen	Before Maceration	After Maceration	Change in Nitrogen Content
Total	2.5	2.9	+0-4
Soluble	2.3	2.5	+0.2
In Suspension	0.2	0-4	+0.2

Thus in this variety there was a 9% increase in soluble nitrogen following maceration of the pulp; in Bulmer's Norman there was also a 9% increase, i.e. from 16-9 mg. to 18-4 mg.; in Kingston Black there was a 12% increase from 1-7 mg. to 1-9 mg. Tavernier and Jacquin (5) reported no change after maceration; they, however, did not specifically estimate soluble nitrogen, but carried out all their nitrogen analyses on juices which had been strained through fine silk.

Nitrogen Changes which take place during the Defecation Process.

These changes are illustrated in Table VI. Thus it is seen for Sweet Alford

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TABLE VI.

NITROGEN CHANGES DURING DEFECATION.

SWEET ALFORD AND RED FOXWHELP.

(Results expressed as mg.N/100 ml. Juice).

Treatment	Nitrogen		Sweet Alford	ALFORD			RED FOXWHELP	XWHELP	
		Freshly Expressed Juice	Clarified Juice, i.e. after Defecation	Change in Nitrogen Content	Percentage Change	Freshly Expressed Juice	Clarified Juice, i.e. after Defecation	Change in Nitrogen Content	Percentage Change
	Total	4.8	2.3	-2.5	52%	20.00	1.6	6.0	-36%
Defecation Only (D)	Soluble	4-4	1.7	7.5-	-61%	2.3	1.3	-1.0	43%
	In Suspension	0.4	9-0	+0.2	+50%	0.3	0.3	+0.1	+50%
Maceration	Total	4.8	2.0	2.8	-28%	9.9	2.0	6.0	-31%
and Defecation (M + D)	Soluble	4.0	1.7	2.3	9%89—	2.5	1.6	6.0	-36%
	In Suspension	8.0	0.3	-0.5	-63%	0.4	0.4	0.0	lil

and Red Foxwhelp, where defecation was successful, that there was a considerable decrease in the soluble nitrogen content of the juices as a result of defecation. The decreases in soluble nitrogen which occurred in the untreated juices, i.e. Controls, to which reference has already been made were also considerable but, as explained in the Discussion, have an entirely different significance.

Changes observed in the nitrogen in suspension were irregular, small in amount and call for no special comment.

Nitrogen Changes Subsequent to Defecation.

For Sweet Alford and Red Foxwhelp, changes in soluble nitrogen during the fermentation of defecated juices from both unmacerated and macerated pulp, i.e. D and M+D, were only slight in extent. In contrast, changes in soluble nitrogen during the fermentation of juices that had not been submitted to defecation, i.e. C and M, were very large. The nitrogen changes for the fermentations of the juices from macerated Sweet Alford pulp, i.e. both M and M+D, are illustrated in Fig. 4.

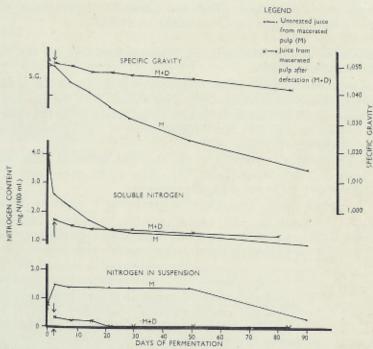


Fig. 4. Fermentations of the Juices from Macerated Pulp of Sweet Alford. Specific Gravity Curves and Changes in Soluble Nitrogen and Nitrogen in Suspension.

The arrow † indicates the day on which the defected juice (M + D) was siphoned off from the brown head and lees.

For comparison the macerated juice will be considered first. In this juice, as illustrated in Fig. 4, there was a large decrease in soluble nitrogen, from 4.0 mg. to 1.4 mg. in the first twenty-one days, as a result of the rapid growth of yeast which occurred; during this time there was a large decrease in specific gravity (15 degrees). Subsequent decreases in soluble nitrogen were small, only 0.4 mg., so that after twenty-one days there was little further

growth of yeast. However, the specific gravity continued to decrease a further 21 degrees over the remaining fifty-nine days.

In contrast to the large change in soluble nitrogen which occurred during the fermentation of the macerated juice, the corresponding changes in the juices which defecated were (as stated above) very small. Thus, in eighty days there was a loss of only 0.5 mg, soluble nitrogen, indicating that very slight growth of yeast took place during this period. Further evidence that growth of yeast was slight is given by (i) the small amounts both of nitrogen in suspension, which is regarded as representative of yeast in suspension, and of nitrogen in the lees, which is representative of yeast in the lees, and by (ii) the slow rate and restricted extent of the fermentation, e.g. a decrease of only 9 degrees of specific gravity during the eighty days as compared with a decrease of 34 degrees in the macerated, non-defecated juice (M), for this period.

It may be observed from Figs. 3 and 4 that if the untreated juices C and M, which did not defecate, had been siphoned into fresh casks at the same time as the treated juice M+D, large amounts of yeasts would still have been left in suspension. Thus the subsequent fermentation of these untreated juices would not have been greatly restricted.

The Effects of these Treatments on Fermentation and on the Stability of the Resulting Ciders.

The main object of the process of defecation is the production of naturally sweet ciders. Thus, the defecation treatment reduces both the rate and extent of the fermentation so that fermentation either ceases while the cider is still naturally sweet or is so slow that artificial control, e.g. by centrifuging, can be exercised effectively.

The effect of the treatments on the rate and extent of fermentation of three juices is illustrated in Table VII. The fourth juice from Kingston Black did not respond to the defecation process.

TABLE VII.

EFFECT OF MACERATION AND DEFECATION ON THE RATE OF FERMENTATION.

BULMER'S NORMAN, SWEET ALFORD AND RED FOXWHELP.

		BULMER'S	NORMAN	SWEET	ALFORD	RED FO	XWHELP
Treatment	,	Rate of Fermentation at 25° C. in the laboratory	Time to Ferment to S.G. 1,025 in the Ciderhouse	Rate of Fermentation at 25° C. in the laboratory	Time to Ferment to S.G. 1,025 in the Cklerhouse	Rate of Fermentation at 25° C. in the laboratory	Time to Ferment to S.G. 1,025 in the Ciderhouse
No Treatment * (C)		7-8	7 days	1.1	48 days	0-7	54 days
Defecation only+ (D)		4.4	17 days	-	90 days	†	†
Maceration only*		7-8	6 days	1+3	51 days	0.8	101 days
Maceration & Defect (M + D)	ion+	2.3	30 days	0-6	180 days	0-3	171 days

^{*} i.e. the fresh juices.

It will be seen that the preliminary defectaion treatment has greatly reduced the rate of fermentation. In each of the three varieties the rates of fermentation of the defecated juices, i.e. D and M + D, were slower than those of the corresponding non-defecated juices C and M. The effects of maceration on the rates of fermentation were not consistent throughout the three series so that no definite conclusion can be drawn at present.

The extent of fermentation in the defecated juices varied considerably. Only the defecated juice of Red Foxwhelp ceased fermentation while still naturally sweet and this juice commenced fermentation with the lowest soluble nitrogen content (1.2 mg.) of any of the varieties. Although this juice after maceration plus defecation eventually fermented to dryness, this did not occur until after ten months, whereas the untreated juice, i.e. the Control, was dry after six months. In contrast, both the defecated juices of Bulmer's Norman fermented rapidly to dryness. However, in the juice of this variety the initial soluble nitrogen (and probably other yeast nutrients also) was large and presumably sufficient remained after defecation to support growth of yeast in quantity, so that fermentation was rapid. With Sweet Alford the juice after maceration plus defecation developed sickness at specific gravity 1,023 and fermented to dryness; this juice after defecation only, which had reached S.G. 1,008 at this time, did not develop the disorder and fermented to dryness normally. It was observed that the titratable acidity of the cider which developed the disorder (i.e. M + D) remained much lower than the titratable acidities of the other three ciders of this variety. The development of the bacteria would probably have been inhibited if this cider from macerated and defecated juice had been blended earlier with high-acid cider, as in the normal commercial treatment of low-acid juices. This, in fact was not done in these experiments since the ciders were kept deliberately as single varieties in order that their individual fermentations could be studied.

The Effects of these Treatments on the Quality of the Ciders.

The ciders will be referred to in this section according to the treatments originally given to the juices from which they were made, e.g. the Control cider refers to a cider resulting from the fermentation of untreated juice. Similarly the Macerated and Defecated cider refers to the cider made from the defecated juice obtained after maceration of the pulp.

Only the ciders made from Sweet Alford and Red Foxwhelp, i.e. those in which successful defecation occurred, were sampled for bottling (see Experimental, Cider-Making Procedure).

The quality of each cider was assessed by the procedure reported elsewhere (12). Only a summary of the most important features found in each cider will be given.

Naturally Conditioned Ciders.

It was difficult to assess the quality of the naturally conditioned ciders since, with the exception of the Macerated and Defecated cider from Sweet Alford, which was "sick," none of these ciders developed any perceptible degree of carbonation. This lack of conditioning, even in the sweetened ciders was probably due to the fact that they were bottled in the late summer; bottling of the ciders of each variety was delayed until the Macerated and Defecated cider of that variety had fermented to S.G. 1,025.

⁺ i.e. juices which have responded to the defecation process.

[†] Still at S.G. 1,040 one year after pressing the pulp. S.G. of juice before defecation was 1,053.

Artificially Carbonated (Sterile-filtered) Ciders.

The uniform carbonation of the sterile-filtered ciders simplified the

assessment of their quality considerably.

The Control ciders of Red Foxwhelp, both unsweetened (S.G. 1,003.5), and sweetened (S.G. 1,022) had a peculiar resinous aroma which persisted to a smaller extent in the flavour, spoiling an otherwise pleasant and fruity cider. The resinous character, however, was not found in the Defecated cider (S.G. 1,041) of this variety which, although high in acid and tannin, also had a high natural sugar content so that the cider was balanced in flavour. This clean, fruity, pleasant cider was given the highest figure of merit of the ciders made from Red Foxwhelp. All the ciders made from the macerated pulp of Red Foxwhelp possessed an intense, almost quinine-like bitterness which was unpleasant. The unsweetened Macerated cider (S.G. 1,006.5) possessed a fruity aroma, while the aroma of the sweetened cider (S.G. 1,021.5) was slightly resinous. The Macerated and Defecated cider (S.G. 1,021.5) was unpleasant since it had both the resinous aroma and intense bitter flavour.

All but one of the ciders made from Sweet Alford were clean in aroma and flavour. The Control cider unsweetened (S.G. 1,001) was a pleasant, dry cider, whereas the same cider sweetened (S.G. 1,021) was in addition, fruity and typical of the variety. The effect of sweetening in improving the flavour was even more marked in the Defecated ciders, where the unsweetened cider (S.G. 1,001) had little body or character but when sweetened (S.G. 1,021) possessed these qualities in good measure comparable with the sweetened Control cider. The unsweetened Macerated cider (S.G. 1,003.5) was given a slightly greater figure of merit than any other cider made from Sweet Alford, having more acidity, a pleasant tannin bitterness and fruity character; when sweetened (S.G. 1,021) it was well balanced in flavour. The quality of the Macerated and Defecated cider (S.G. 1,021) could not be assessed since the characteristic aroma and flavour of "sickness" was present in slight amount. This cider must have been slightly "sick" when it was sterile-filtered since there were no signs of bacterial fermentation having taken place in the bottle.

Thus the Control ciders, both sweetened and unsweetened, from Sweet Alford were of high quality, whereas the Control ciders from Red Foxwhelp were poor. Maceration of the Sweet Alford pulp apparently improved the quality of the cider but with Red Foxwhelp the same treatment gave a cider inferior to the Control. The quality of the ciders made from macerated and defecated juices, i.e. M + D, could not be assessed, since this cider from Sweet Alford was slightly "sick" and the corresponding cider from Red Foxwhelp had the resinous quality of the fresh juice combined with the intense bitterness due to maceration of the pulp. Defecation of the juice from freshly-milled Sweet Alford pulp gave a cider which, after sweetening, was of high quality; the same cider from Red Foxwhelp was naturally sweet and better than the

corresponding Control cider.

DISCUSSION.

In this present work, four typical varieties of cider apples have been studied in relation to their behaviour during the processes of Maceration and Defecation. The results will be summarised first, and then followed by a discussion in more detail.

(a) The four varieties varied in the extent to which they responded to the two treatments. (b) The process of maceration increased the pectin content of the juice, the increase varying according to the variety of apple. The varieties which showed the largest increase of juice pectin due to maceration also clarified most rapidly during the subsequent defection.

(c) Successful defecation was accompanied by complete precipitation of

pectin from solution.

(d) During the course of defecation there was a considerable reduction of soluble nitrogen in the juice. The rates and extents of the subsequent fermentations of the defecated juices showed some relation to the changes in soluble nitrogen that took place during defecation.

(a) Defecation did not occur in any of the four varieties without the addition of salts. The defecated juices, i.e. D and M + D treatments, of the three varieties, Bulmer's Norman, Sweet Alford and Red Foxwhelp showed rapid clarification; the fourth variety did not clarify satisfactorily. Warcollier (4) and Tavernier (5) have shown in this connection that the maturity of the fruit as well as the variety is also important. Thus, it is not possible to say at present whether these varieties will behave in the same manner each year.

(b) For the three varieties in which pectin changes were followed, maceration caused an increase in the pectin content of the expressed juice; the increase varied according to the variety. It was found that where the pectin increase was greatest and of the order of 50%, as in Sweet Alford and Red Foxwhelp, the subsequent clarification of the macerated and defecated juices was satisfactory. This would suggest that there was an increase in the pectase content of the juice accompanying the large increase in pectin and this is further borne out by the more rapid removal of pectin which occurred in the juice prepared from macerated pulp as compared with juice from unmacerated pulp (Table II). The increase in pectin may itself be due to the action of the enzyme pectase during maceration.

(c) Successful defecation was characterised by the rapid formation of a brown, pectin-jelly head and the complete and rapid removal of soluble pectin from solution. For example, pectin was no longer detectable in the macerated

and defecated juice from Sweet Alford after two days.

It is interesting to compare this process, carried out under controlled conditions, with the phenomenon of "dropping bright" which occurs occasionally in the normal course of cider-making. In this phenomenon, which has been discussed in an early paper by Charley (17), a pectin clot is formed, giving rise to a brilliantly clear juice, which, if racked, ferments very slowly and may cease fermenting while still naturally sweet. It may be supposed that such apple juices contain large amounts of pectase and sufficient calcium ions to precipitate the pectin as a pectate gel. In a normal fermentation a gradual clarification usually occurs and has been found in other experiments to be accompanied by a slow removal of pectin. The influence of pH and of the concentrations of salts in such clarifications will require further investigation, for it is evident from the present experiments that addition of defecating salts does accelerate the course of juice clarification (Table IV).

(d) The soluble nitrogen content of the juice is one of the main factors determining the course of the subsequent fermentation. As emphasised earlier, one of the main effects of defecation is the removal of nitrogen from the juice. In fact, the percentage removal of soluble nitrogen was very great and of the order of 50% in the defecated juices of Sweet Alford and Red Foxwhelp. Consequently the amount of soluble nitrogen remaining for yeast growth in the defecated juice was considerably reduced. For example, when the percentage removal of soluble nitrogen during defecation was greatest (Sweet Alford and Red Foxwhelp) there was a very considerable restriction of fermentation in the defecated juices; in Bulmer's Norman where the percentage removal was less, although the fermentation was restricted compared with the Control of that variety, the juice fermented to dryness in forty days. The importance of the amount of nitrogen still remaining in the defecated juices has been discussed by Tavernier (18) who states that if the ratio

Total Nitrogen mg./litre Total Reducing Sugars g./litre for a defecated juice is not greater than 0.4,

then the fermentation will cease while natural sugar still remains. For Bulmer's Norman the clarified juices after defecation fermented rapidly to dryness and this ratio for the M + D treatment was 1.2. For Sweet Alford and Red Foxwhelp, there was considerable restriction of fermentation, and the ratio for the same treatment, i.e. M + D, was 0.18. This ratio for the defecated juice of Red Foxwhelp which eventually ceased fermentation while naturally sweet was 0.15. It must be emphasised however, that the ratio for the Control juices of Sweet Alford and Red Foxwhelp were themselves only 0.4 and 0.23 respectively. While it is true that a juice with a ratio greater than 0.4 may not remain naturally sweet, these experiments show that the converse is not necessarily true since the juices with a ratio less than 0.4 (with the exception of the defecated Red Foxwhelp juice) whether defecated or not, did not remain naturally sweet. Thus the differences in the extent of fermentation observed even in the juices in which this ratio was low, cannot be explained entirely by the nitrogen contents of the defecated juices. In fact, the variations in behaviour showed by the defecated juices of Sweet Alford and Red Foxwhelp were not entirely consistent with the amounts of nitrogen remaining in the juice. Further investigation may show that only certain nitrogenous constituents may be concerned in this connexion and or that deficiencies of other yeast nutrients may also be concerned.

The mechanism whereby soluble nitrogen was removed from the juice was not investigated specifically in these experiments and must be the subject of further research. Tavernier and Jacquin (5) state that defecation converts the greater part of the soluble proteins and polypeptides of the juice into insoluble compounds which are removed in the brown, pectin-jelly head. They state that these nitrogenous constituents are essential for yeast growth. and that in consequence, such a juice could only ferment very slowly and often ceased to ferment while the juice was still naturally sweet. However, Thorne (19) and other workers who have investigated the assimilation of different nitrogenous constituents by yeast, have shown that the yeasts they were examining, utilised single amino acids more readily than simple peptides and much more readily than polypeptides. A possible mechanism suggested by the work reported in this present paper is as follows: When the juice is not submitted to defecation, a rapid growth of yeast takes place: such growth of yeast did in fact occur during the "lag" period of the normal fermentation of these untreated juices, i.e. the Controls, depleting the juices not only of nitrogen but also of other yeast nutrients (see Burroughs and Challinor (20)). In the defecated juices any yeast crop would be removed in the brown jelly head or lees. It has been generally assumed that the brown jelly head plays a purely mechanical part in removing the suspended material,

whether it consists of "precipitated polypeptides" or of yeast cells, but chemical adsorption between the precipitated pectin complex and the amino acids of the juice may also occur.

The effects of these different treatments on the quality of the ciders made from Sweet Alford and Red Foxwhelp have already been discussed in an earlier section of this paper and only a brief summary of the more important findings will be made here. For these two varieties, the ciders made after defecation of the juices were of high quality; in particular the naturally-sweet cider from this treatment of Red Foxwhelp juice was of much higher quality than the corresponding untreated, Control cider. From the limited experience obtained in these experiments involving maceration in cider-making it is not possible to generalise about its effect on the final cider, for while anaerobic maceration of the low-acid, Sweet Alford pulp was beneficial, maceration of the highly acid, bittersharp, Red Foxwhelp pulp was detrimental since the

tannin bitterness was unpleasantly emphasised.

The production of a naturally sweet cider by preliminary defecation of the juice demands very close control both of the juice and of the fermentation at all stages and a clear understanding of the processes involved (see also Warcollier (4)). To facilitate this control, the cider-making procedure and factory lay-out employed must be specially designed, if the process is to be operated economically on a large scale. Thus, the milled pulp should preferably be allowed to fall into small tanks above the presses for maceration. After maceration the pulp should then fall by gravity to the cheese-building platform. Similarly, the tanks for the defecation process should be totally enclosed, and fitted with observation windows and attemperator coils, so that the temperature of the juice undergoing defecation can be controlled to ensure that pectin clotting is complete before fermentation commences.

Irrespective of the quality of the resulting ciders or the degree of control involved in carrying out the processes, this study of maceration and defecation and the subsequent fermentations has given further insight into fundamental aspects of cider-making in general. For a more complete understanding of the mechanism of the defecation process, it will be necessary to supplement these results by qualitative examination of the types of nitrogenous constituents present in the juice initially and after the process of defecation has occurred.

SUMMARY.

1. An investigation has been made of the effects of maceration and defecation on the juices of four varieties of cider apples.

The four varieties varied in the extent to which they responded to the two treatments. Three of the four juices clarified and responded well to the treatment.

Maceration increased the pectin and pectase contents of the expressed juices and thus facilitated subsequent defecation.

Successful defecation was accompanied by complete precipitation of pectin from solution.

The soluble nitrogen contents of the juices which responded to

defecation were thereby greatly reduced.

The rates and extents of the subsequent fermentations of the defecated juices showed some relation to the changes in soluble nitrogen that occurred as the result of defecation, although the results suggest that factors other than soluble nitrogen may also be concerned.

- 7. An assessment has been made of the quality of the ciders produced after these treatments. Ciders made from defecated juices were of high quality. The effects of maceration on the quality of the ciders varied with the variety.
- 8. An indication has been given of some of the practical cider-making aspects of these two processes.

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REFERENCES.

- Challinor, S. W. Ann. Rept. Long Ashton Res. Stn., 1948, 216.
 Charley, V. L. S. Ibid., 1937, 160.
 Pollard, A. Ibid., 1948, 222.

- "The Principles and Practice of Cider-Making," a translation by Dr. V. L. S. Charley of "La Cidrerie" by Prof. G. Warcollier. Chapters VI and IX.
- TAVERNIER, J. and JACQUIN, P. Comptes rend., Acad. Sci., 1946, 222, 416. CHARLEY, V. L. S. Ann. Rept. Long Ashton Res. Stn., 1935, 138.

- Kieser, M. E., Pollard, A. and Stone, A. M. Ibid., 1948, 228.
 Pollard, A. and Kieser, M. E. J. Sci. Food Agric., 1951, 2, 30.
- (9) LE CORVAISIER, H. Chim. et Ind., 1946, 56, 382.
- (10) Bejambes, M. and Tavernier, J. Bull. Assoc. Chim. Sucr., 1945, 62, 135.
- Burroughs, L. F. Ann. Rept. Long Ashton Res. Stn., 1946, 127.
 Challing, S. W. and Burroughs, L. F. Ibid., 1947, 172.
- (13) Methods of Analysis of A.O.A.C. (6th Edition), 1945, Chapter XV.
- (14) Burroughs, L. F. and Challinger, S. W. Ann. Rept. Long Ashton Res. Stn., 1949, 115.
- (15) HINTON, C. L. Fruit Pectins, D.S.I.R. Special Report, No. 48, 1939, Part I.
- (16) CHALLINOR, S. W. and BURROUGHS, L. F. Ann. Rept. Long Ashton Res. Stn., 1948, 182.
- (17) CHARLEY, V. L. S. Ibid., 1934, 217.
- (18) JACQUIN, P. and TAVERNIER, J. Extract du procès-verbal de la Seance, Académie D'Agriculture de France, 24th January, 1951.
- THORNE, R. S. W. Wallerstein Labts. Communs., 1950, 13, 319.
- (20) Burroughs, L. F. and Challinor, S. W. Ann. Rept. Long Ashton Res. Stn., 1950, 161.



Fig. 1. Typical brown, pectin-jelly head formed during successful defecation,



Fig. 2. Typical "white head" formed during the early stages of fermentation of juices that did not defecate.