

## Chapter 43

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# Characterisation of the pot ale profile from a malt whisky distillery

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### Introduction

The Scotch Whisky industry is one of the most productive industries in Scotland, second only to oil and gas. Scotch Whisky is one of the few products defined by UK law. Distilleries are often located in scenic areas and their importance is magnified in providing an economic backbone to local communities. The environmental impact of all industrial practices are of paramount importance nowadays, and it is especially important that the Scotch Whisky industry is observed to have minimal deleterious influences on the environment.

During the production of Scotch malt whisky, a large volume of pot ale is produced. Pot ale is a nutrient rich co-product, the residue of fermented wort left in a pot still after the distillation process. (See figure 1) Pot ale is very concentrated and a relatively large volume is produced. For example, for every litre of whisky made, eight litres of pot ale is produced (Mohana, Acharya and Madamwar, 2008). Pot ale is a very complex, caramelised and cumbersome organic turbid liquid residue. It is characterised by a high COD/BOD, possesses dark colour and has a high solids content. In volume and strength pot ale represents the most significant wastewater from the distilling process (Toduka, Fujiwara and

Kida, 1999) The composition of pot ale needs to be analysed so that, following its treatment and discharge into watercourses, the environmental impact can be minimised. Many distilleries are located in remote locations in Scotland which need to be protected against any aquatic and terrestrial pollution. This is safeguarded by The Scottish Environmental Protection Agency (SEPA) which ensures effluent emissions comply with legislative requirements. Distilleries' priorities are to discharge effluents that comply with the legal limits of parameters such as BOD, COD and copper content. Distilleries are now additionally looking to find economically viable options to dispose of pot ale. In order to do this the components of pot ale need to be identified to ensure the correct biological treatments provide optimum output and reduce levels of final unfavourable constituents.

This chapter describes research focusing on pot ale from a single distillery, but the findings are pertinent environmentally and economically to the Scotch whisky industry generally. The research employed different techniques to analyse the following constituents of pot ale: BOD, COD, pH, total volatile acids and copper content.

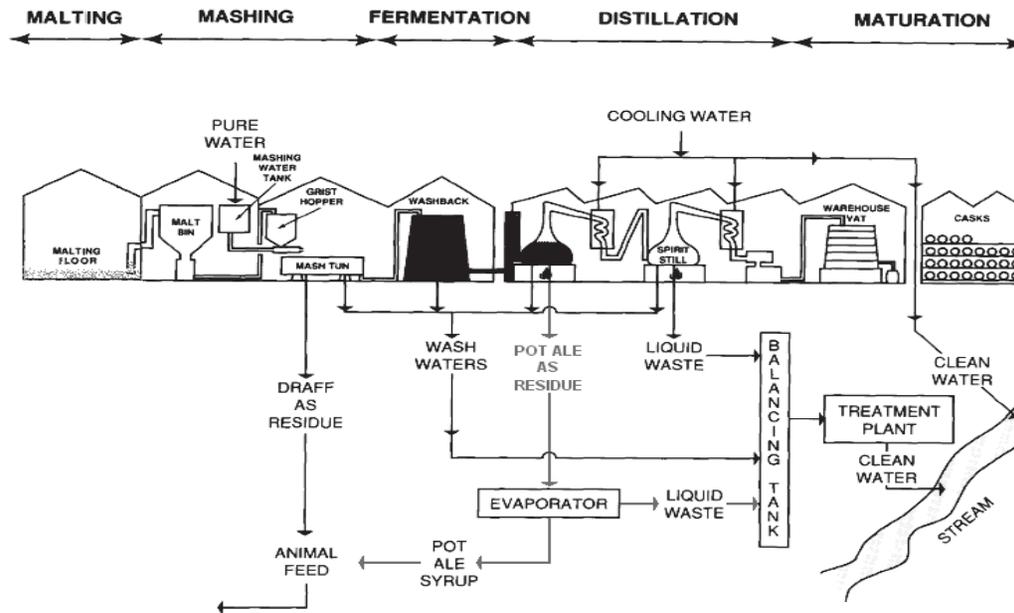


Figure 1. Residues and co-products from the whisky making process (McBoyle 1996)

The aim of the work was to assess (using different analytical techniques) variability in these constituents in pot ale streams from a malt whisky distillery over time.

### Materials & methods

Samples were taken weekly from a malt whisky distillery from both the wash still and an external pot ale tank. Comparison between the weekly pot ale compositions could therefore be established. (Refer to figures 2 and 3) In addition, extra samples were taken within the week from the external tank, to test if there were variations within the week.

To characterise pot ale, various analytical techniques were utilised. The organic content of pot ale was examined by measuring the Chemical Oxygen Demand (COD) and the Biological Oxygen Demand (BOD). If pot ale with high COD/BOD values enters the water system, microorganisms metabolise the organic materials present whilst purifying the water, and oxygen in the water will be consumed. Oxygen may not be replaced fast enough to sustain

aquatic life so the waterways will deteriorate as a result of the death of aerobic organisms. Thus there is a need to restrict COD/BOD levels (Ansa-Asare, Marr and Cresser 2000) in residues discharged from distilleries, such as pot ale.

COD provides a measure of the oxygen equivalent to that portion of the organic matter in waste water susceptible to oxidation by a strong chemical oxidant. Diluted pot ale was added to a pre-prepared COD vial of reagent solution. The samples were placed in a heating block for 2 hours at 150°C then analysed spectrophotometrically.

BOD relates to the quantity of oxygen (ppm) consumed in waste water by microorganisms measured over a 5 day period. This was completed in defined conditions at 20°C in the dark. An oxygen probe sensor measured the percentage of oxygen in the sample. In order to establish the optimal working range the samples were appropriately diluted.

The copper content of pot ale was measured by ion chromatography (IC) and atomic absorption spectroscopy (AAS). Copper stills are used in the distillation stage. Transfer occurs between the hot still and the refluxing liquid resulting

in copper being present in the pot ale. Pot ale has traditionally been used as an ingredient of animal feeds. However, this may be poisonous to sheep as they cannot metabolise copper. Pot ale samples for atomic absorption spectroscopic analysis were pre-digested with nitric acid whereas samples for ion chromatography analysis were pre-filtered to give the copper concentration in the liquid fraction, so a comparison could be made with the AAS results for the complete fraction.

Pot ale contains volatile acids which decrease the effluent pH. If the pot ale sample is fresh, the acetic acid concentration will be ~1,000mg/l, but if the sample is sour it will be ~10,000mg/l. This is due to bacterial metabolic activity (IBD General Certificate of Distilling 2009). Total acids present in pot ale samples were measured by titration and the individual volatile acids of interest – acetic, lactic and propanoic acid by high performance liquid chromatography (HPLC).

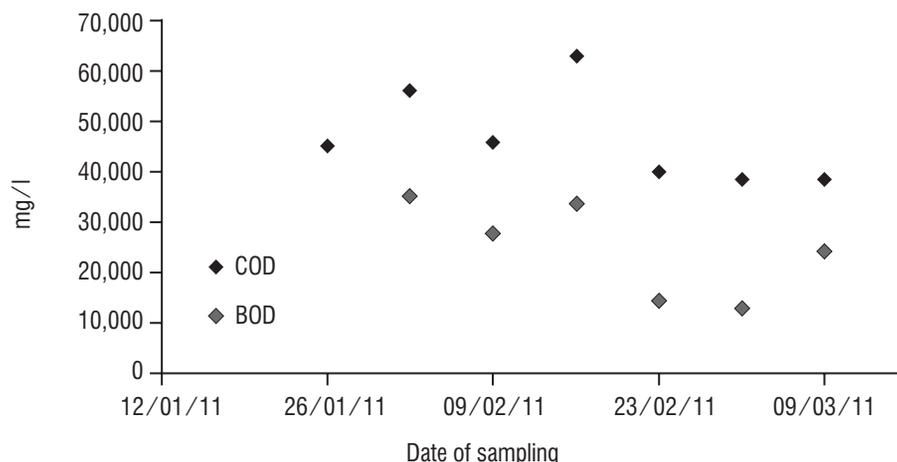
## Results and discussion

BOD and COD levels fluctuated significantly throughout the sampling period, as shown in figure 4. The peaks and troughs appear to be at the same positions so the BOD and COD measurements follow the same pattern. More fluctuations are apparent with the external tank samples which could be due to tank heterogeneity (the agitator being out of order) so the sample was unrepresentative. The BOD had an average value of 24,908mg/l with a range of 12,951 to 35,327mg/l. The COD average was 46,854mg/l with a range of 38,476 to 62,948mg/l. The difference between BOD and COD measurements were similar for the weekly sampling period, demonstrated by a correlation coefficient of 0.71. Both BOD and COD levels, which followed similar patterns, exhibited large variations.

A titration technique was used to determine the total acids present in the pot ale samples and



Figures 2 and 3. Wash still (left) and external tank (right)

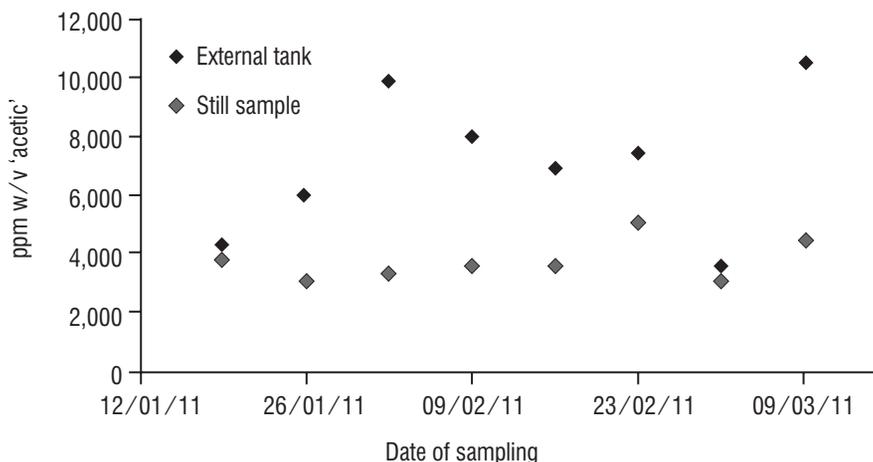


**Figure 4.** BOD and COD levels in pot ale from a distillery external tank over time

these were also observed to fluctuate over time. The external tank samples contained more acids than the still sample which is due to microbial auto-fermentation (see figure 5). Total volatile acidity values in the pot still samples varied between 3000 to 5040ppm, with an average of 3720ppm. In comparison the external tank measured fluctuated from 3600 to 10560ppm, with an average of 7079ppm which is nearly double the total acids present in the still sample. The pot still samples were more constant over time, while the external tank volatile acidity had a large variation due to tank heterogeneity and unrepresentative sampling. There was no correlation between the external tank and still samples for the total acids present (correlation coefficient of 0.19).

Individual acids analysed included acetic, lactic and propanoic acids with respective analyses of > 1000mg/L, 120mg/L and 85mg/l. Acetic acid is high due to microbial conversion from lactic acid during fermentation. Small fluctuations were seen for each sample. Figure 6 shows that the acids varied proportionally (as one acid increased the others also increased).

Regarding pH, small fluctuations occurred at the same points with the sample of pot ale from the still possessing a higher pH for each week analysed. This is due to higher total volatile acidity observed in the external tank which had a range of pH 3.25 to 3.92 and average of 3.54. The still samples were slightly higher with a range of pH 3.34 to 4.13 and an average of 3.81.



**Figure 5.** Total volatile acids in pot ale from external tank and still samples over time

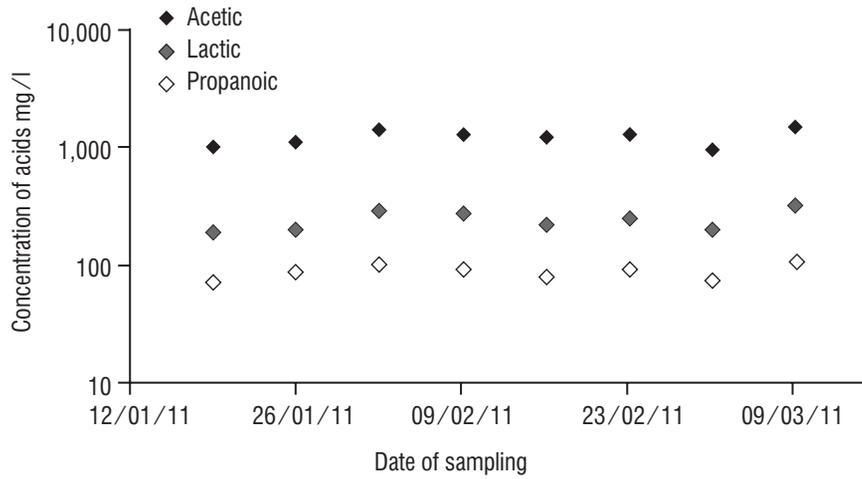


Figure 6. Individual acids in pot ale samples from a distillery external tank over time

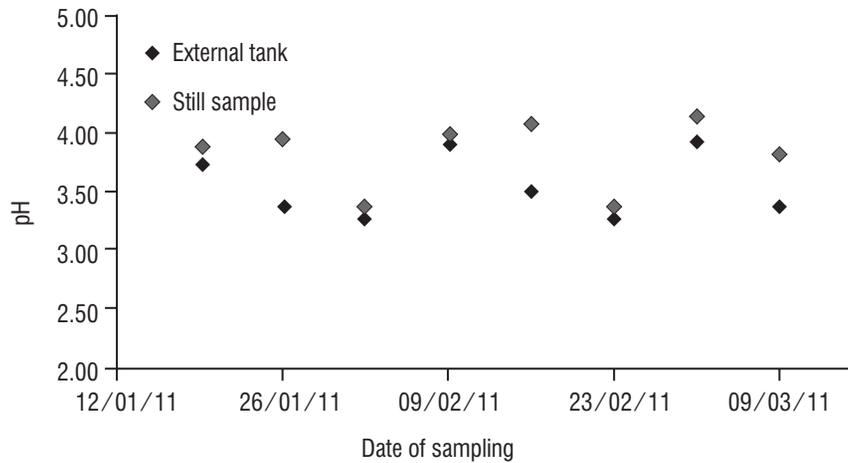


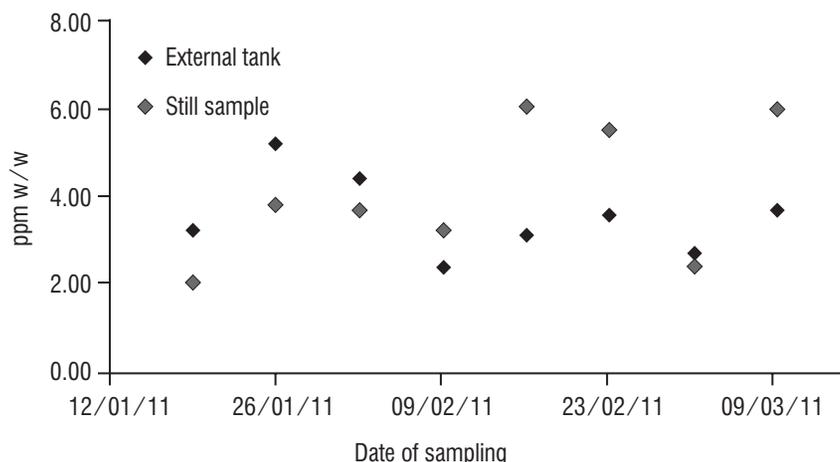
Figure 7. Variation of pH for in pot ale samples from distillery external tank and still over time

With regard to copper analysis, no relationship existed between the samples and copper content (correlation coefficient 0.02). The results varied, with the external tank or still sample occasionally containing more copper. Copper levels in pot ale fluctuated from 2-6ppm throughout the 8 week sampling period. Atomic absorption spectrophotometry (AAS) measures the copper present in the whole pot ale sample whilst ion chromatography measures the soluble copper present in sample filtrates. Figures 8 and 9 show that no relationship was found between the analytical approaches adopted, although more copper was generally found by AAS, which analyses total (bound and soluble) copper in pot ale .

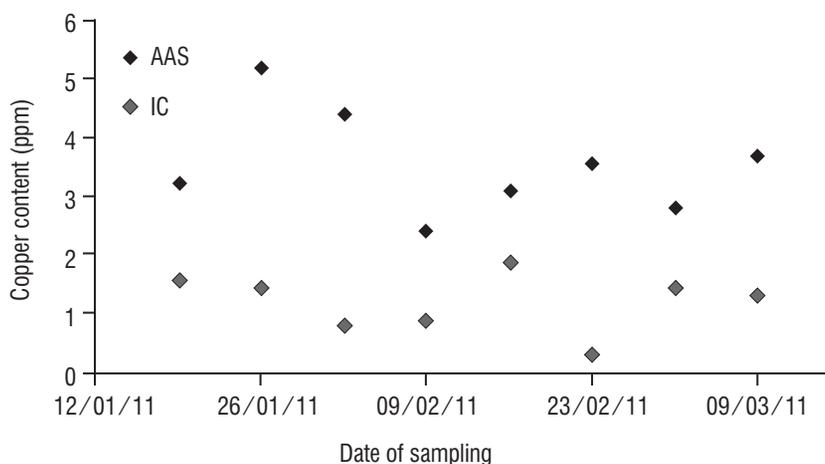
## Conclusion

This study has revealed significant inconsistencies in distillery pot ale composition throughout an 8 week sampling period. Care was taken to ensure that replicate samples were removed from the same position each time from both the pot still and the external storage tank. As part of the test regime replicates were taken and averages calculated. Each time the replicates had similar results.

The analytical findings indicate that compositional variation in pot ale was due to inherent differences in pot ale composition, rather than sampling techniques. One of the sources of error may have been that the external



**Figure 8.** Copper content in pot ale from distillery samples by AAS technique



**Figure 9.** Comparison of copper content in distillery pot ale samples by AAS and IC techniques

tank stirrer was not operational and further work would be required to obtain more homogeneous samples. However, large variations in BOD and COD were observed in the still samples which were unaffected by any lack of stirring.

Whisky raw material ingredients and production processes were constant throughout the sampling period and it was initially assumed that pot ale would not vary during sampling. However, variability in the analysed parameters was observed, although the underlying reasons for this remain unclear. Further research will verify if processing, storage or transportation may account for pot ale compositional differences.

This study has highlighted the importance of precise analyses of distillery pot ale residues destined for further treatment or for co-products utilisation in animal feeds.

### Acknowledgements

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