

Table S2. Peak information, relative intensity and *p*-value of volatile compounds by ethyl acetate extraction

| Compound name | RI ^a | Quantitative m/z | Relative peak height (mean ± SD) ^b | | | | | Reliability ^d | <i>p</i> -value ^c | |
|---|-----------------|---------------------|---|-------------|-------------|--------------|-------------|--------------------------|------------------------------|-----------------------------|
| | | | 0 week | 18 weeks | | | | | Control vs. LAB + EtOH | Control vs. Acid + Yeast |
| | | | | Control | LAB + EtOH | Acid + Yeast | Acid + EtOH | | | |
| 1-Propanol | | 59 | 0.002±0.00 | 0.208±0.004 | 0.111±0.004 | 0.189±0.011 | 0.112±0.004 | C | < 0.01 * | 0.074 |
| 2-Methyl-1-propanol (Isobutyl alcohol) | | 74 | 0.019±0.00 | 0.168±0.006 | 0.023±0.001 | 0.131±0.009 | 0.021±0.000 | A, C | < 0.01 * | 0.010 |
| 1-Butanol | | 56 | 0.004±0.00 | 1.596±0.016 | 0.006±0.000 | 1.672±0.131 | 0.005±0.001 | A | < 0.01 * | 0.502 |
| 3-Methyl-1-butanol (Isoamyl alcohol) | 1205.48 | 55 | 0.072±0.00 | 0.994±0.081 | 0.093±0.009 | 0.788±0.069 | 0.078±0.005 | A, C | < 0.01 * | 0.052 |
| 3-Hydroxy-2-butanone (Acetoin) | 1274.97 | 88 | 0.002±0.00 | 0.675±0.021 | 0.007±0.001 | 1.037±0.041 | 0.003±0.000 | A, B, C | < 0.01 * | < 0.01 * |
| 1-Hydroxy-2-propanone (Acetol) | 1287.97 | 74 | 0.002±0.00 | 0.012±0.001 | 0.012±0.001 | 0.016±0.001 | 0.016±0.001 | A, B, C | 0.501 | < 0.01 * |
| Ethyl lactate | 1334.19 | 75 | 0.002±0.00 | 1.078±0.015 | 1.109±0.024 | 1.216±0.067 | 1.082±0.104 | A, B, C | 0.204 | 0.048 |
| 1-Hydroxy-2-butanone | 1360.78 | 57 | 0.002±0.00 | 0.105±0.004 | 0.020±0.001 | 0.120±0.006 | 0.015±0.001 | C | < 0.01 * | 0.040 |
| Acetic acid | 1419.88 | 59 | 0.003±0.00 | 0.061±0.002 | 0.057±0.004 | 0.052±0.008 | 0.055±0.008 | A, B, C | 0.158 | 0.154 |
| 3-(Methylthio)-1-propanal (Methional) | 1440.77 | 57 | 0.025±0.00 | 0.021±0.000 | 0.022±0.000 | 0.021±0.000 | 0.021±0.001 | A, B, C | < 0.01 * | 0.191 |
| 2-Furancarboxaldehyde (Furfural) | 1448.22 | 96 | 0.002±0.00 | 0.067±0.002 | 0.028±0.001 | 0.077±0.006 | 0.033±0.007 | A, B, C | < 0.01 * | 0.097 |
| 2,3-Butanediol | 1525.25 | 57 | 0.002±0.00 | 0.460±0.053 | 0.032±0.000 | 0.447±0.041 | 0.027±0.003 | A, B, C | < 0.01 * | 0.808 |
| 2-Methylpropanoic acid (Isobutyric acid) | 1553.84 | 73 | 0.012±0.00 | 0.135±0.005 | 0.147±0.008 | 0.123±0.004 | 0.128±0.021 | B, C | 0.144 | 0.053 |
| Dihydro-5-methyl-2(3 <i>H</i>)-furanone (<i>gamma</i> -Valerolactone) | 1570.28 | 56 | 0.002±0.00 | 0.016±0.001 | 0.002±0.000 | 0.020±0.002 | 0.002±0.001 | A, C | < 0.01 * | 0.045 |
| Dihydro-2(3 <i>H</i>)-Furanone (<i>gamma</i> -Butyrolactone) | 1606.45 | 86 | 0.002±0.00 | 0.028±0.001 | 0.019±0.001 | 0.019±0.002 | 0.014±0.001 | A, B, C | < 0.01 * | < 0.01 * |
| Butanoic acid (Butyric acid) | 1612.62 | 60 | 0.004±0.00 | 0.067±0.003 | 0.043±0.003 | 0.054±0.001 | 0.043±0.002 | B, C | < 0.01 * | < 0.01 * |
| Phenylacetaldehyde | 1624.42 | 91 | 0.004±0.00 | 0.068±0.002 | 0.058±0.005 | 0.069±0.003 | 0.057±0.003 | A, C | 0.056 | 0.652 |
| 2-Furanmethanol (Furfuryl alcohol) | 1643.06 | 98 | 0.002±0.00 | 0.127±0.011 | 0.06±0.005 | 0.103±0.009 | 0.065±0.021 | A, B, C | < 0.01 * | 0.077 |
| 3-Methylbutanoic acid (Isovaleric acid) | 1654.63 | 60 | 0.167±0.010 | 0.499±0.027 | 0.561±0.032 | 0.458±0.033 | 0.530±0.039 | A, B, C | 0.109 | 0.242 |
| 2-Methylbutanoic acid | 1655.64 | 74 | 0.054±0.00 | 0.147±0.005 | 0.162±0.002 | 0.139±0.009 | 0.153±0.016 | A, B, C | 0.012 * | 0.313 |
| 5-Ethyl-2(3 <i>H</i>)-furanone (<i>gamma</i> -hexalactone) | 1679.10 | 85 | 0.000±0.00 | 0.006±0.000 | 0.003±0.000 | 0.005±0.000 | 0.003±0.000 | A, B, C | < 0.01 * | < 0.01 * |
| 3-Methyl-2(5 <i>H</i>)-furanone | 1695.06 | 69 | 0.002±0.00 | 0.055±0.001 | 0.002±0.000 | 0.063±0.008 | 0.002±0.001 | B, C | < 0.01 * | 0.309 |
| 3-(Methylthio)-1-propanol (Methionol) | 1698.43 | 106 | 0.017±0.00 | 0.173±0.006 | 0.045±0.001 | 0.153±0.010 | 0.040±0.002 | A, B, C | < 0.01 * | 0.075 |
| 2(5 <i>H</i>)-furanone | 1730.55 | 55 | 0.002±0.00 | 0.012±0.001 | 0.012±0.000 | 0.014±0.001 | 0.014±0.000 | A, C | 0.419 | 0.158 |
| 2-Butenoic acid | 1754.92 | 86 | 0.002±0.00 | 0.016±0.001 | 0.006±0.001 | 0.020±0.004 | 0.006±0.000 | B, C | < 0.01 * | 0.268 |
| 2-Hydroxy-3-methyl-2-cyclopenten-1-one (Cyclotene) | 1805.57 | 112 | 0.002±0.00 | 0.015±0.000 | 0.010±0.001 | 0.008±0.001 | 0.004±0.000 | B, C | < 0.01 * | < 0.01 * |
| 2-Methyl-2-butenic acid | 1827.69 | 55 | 0.004±0.00 | 0.014±0.001 | 0.016±0.000 | 0.013±0.001 | 0.015±0.002 | A, B, C | < 0.01 * | 0.428 |
| Hexanoic acid (Caproic acid) | 1829.06 | 60 | 0.016±0.00 | 0.012±0.000 | 0.013±0.001 | 0.010±0.002 | 0.012±0.001 | B, C | 0.193 | 0.153 |
| 2-Phenylethyl alcohol | 1891.20 | 91 | 0.014±0.00 | 1.079±0.027 | 0.075±0.004 | 0.930±0.119 | 0.053±0.002 | A, B, C | < 0.01 * | 0.159 |
| 3-Hydroxy-2-methyl-4 <i>H</i> -pyran-4-one (Maltol) | 1938.84 | 126 | 0.013±0.00 | 0.272±0.006 | 0.281±0.014 | 0.255±0.017 | 0.294±0.016 | A, B, C | 0.434 | 0.274 |
| 2-Acetylpyrrole | 1947.68 | 94 | 0.002±0.00 | 0.012±0.000 | 0.015±0.001 | 0.012±0.001 | 0.016±0.001 | A, B, C | 0.014 * | 0.560 |
| 5-Ethyl-2(3 <i>H</i>)-furanone | 1952.29 | 83 | 0.004±0.00 | 0.017±0.001 | 0.002±0.000 | 0.017±0.001 | 0.002±0.000 | B, C | < 0.01 * | 0.600 |
| (<i>R</i>)-Dihydro-3-hydroxy-4,4-dimethyl-2(3 <i>H</i>)-furanone | 2003.10 | 71 | 0.004±0.00 | 0.091±0.002 | 0.084±0.003 | 0.100±0.003 | 0.100±0.005 | C | 0.055 | 0.021 |
| 2,5-Dimethyl-4-hydroxy-3(2 <i>H</i>)-furanone (HDMF) | 2008.05 | 128 | 0.002±0.00 | 0.013±0.001 | 0.018±0.001 | 0.006±0.001 | 0.009±0.001 | A, B, C | < 0.01 * | < 0.01 * |
| 3,5-Dimethyl-4-heptanone | 2039.48 | 57 | 0.002±0.00 | 0.408±0.006 | 0.014±0.001 | 0.403±0.058 | 0.008±0.000 | C | < 0.01 * | 0.910 |
| 5(or 2)-Ethyl-4-hydroxy-2(or 5)-methyl-3(2 <i>H</i>)-furanone (HEMF) | 2070.65 | 71 | 0.002±0.00 | 0.307±0.011 | 0.011±0.000 | 0.305±0.028 | 0.005±0.001 | A, B | < 0.01 * | 0.950 |

| Compound name | RI ^a | Quantitative ve <i>m/z</i> | Relative peak height (mean ± SD) ^b | | | | | Reliability ^d | <i>p</i> -value ^c | |
|--|-----------------|-------------------------------|---|-------------|-------------|--------------|-------------|--------------------------|------------------------------|-----------------------------|
| | | | 0 week | 18 weeks | | | | | Control vs. LAB + EtOH | Control vs. Acid + Yeast |
| | | | | Control | LAB + EtOH | Acid + Yeast | Acid + EtOH | | | |
| 4-Hydroxy-5-methyl-furanone | 2088.91 | 114 | 0.002±0.00 | 0.122±0.011 | 0.320±0.010 | 0.091±0.012 | 0.303±0.038 | A, B | < 0.01 * | 0.055 |
| Caprolactam | 2144.00 | 55 | 0.010±0.00 | 0.075±0.015 | 0.077±0.006 | 0.092±0.028 | 0.081±0.005 | A, C | 0.912 | 0.507 |
| 2-Methoxy-4-vinylphenol (4-Vinylguaiacol) | 2170.61 | 135 | 0.356±0.00 | 0.961±0.082 | 0.855±0.016 | 0.659±0.071 | 0.771±0.060 | A, B, C | 0.145 | 0.017 |
| 1,2,3-Benzenetriol (Pyrogallol) | 2237.13 | 154 | 0.006±0.00 | 0.100±0.008 | 0.078±0.015 | 0.064±0.024 | 0.087±0.019 | C | 0.147 | 0.111 |
| 3,5-Dihydroxy-6-methyl-2,3-dihydro-4 <i>H</i> -pyran-4-one | 2240.95 | 101 | 0.002±0.00 | 0.029±0.002 | 0.054±0.002 | 0.044±0.041 | 0.048±0.009 | C | < 0.01 * | 0.666 |
| Ethyl hexadecanoate (Ethyl Palmitate) | 2249.03 | 88 | 0.002±0.00 | 0.151±0.012 | 0.117±0.009 | 0.184±0.006 | 0.115±0.011 | C | 0.031 | 0.023 |
| 2-Methoxy-6-methyl-4-pyran-4-one | 2279.45 | 69 | 0.002±0.00 | 0.083±0.004 | 0.043±0.001 | 0.082±0.006 | 0.035±0.005 | C | < 0.01 * | 0.838 |
| 4-Oxopentanoic acid (Levulinic acid) | 2296.21 | 56 | 0.002±0.00 | 0.018±0.002 | 0.025±0.001 | 0.025±0.002 | 0.030±0.002 | A, C | 0.012 * | 0.021 |
| mono-Ethyl succinate | 2353.65 | 101 | 0.002±0.00 | 1.592±0.026 | 0.882±0.024 | 1.665±0.121 | 0.913±0.051 | C | < 0.01 * | 0.449 |
| Benzoic acid | 2422.49 | 105 | 0.002±0.00 | 0.002±0.000 | 0.002±0.000 | 0.002±0.000 | 0.002±0.001 | C | 0.484 | 0.875 |
| Phenylacetic acid | 2530.37 | 91 | 0.002±0.00 | 0.288±0.010 | 0.302±0.010 | 0.269±0.013 | 0.280±0.032 | C | 0.257 | 0.180 |
| 4-Ethyl-2,6-dimethoxyphenol | 2541.07 | 180 | 0.005±0.00 | 0.143±0.013 | 0.173±0.008 | 0.126±0.001 | 0.163±0.014 | A, C | 0.051 | 0.215 |
| <i>n</i> -Hexadecanoic acid (Palmitic acid) | 2883.57 | 73 | 0.484±0.00 | 0.037±0.003 | 0.028±0.001 | 0.038±0.001 | 0.024±0.003 | C | 0.013 * | 0.664 |
| 2-(4-Hydroxyphenyl)ethanol | 2961.10 | 107 | 0.002±0.00 | 0.736±0.029 | 0.069±0.001 | 0.657±0.053 | 0.031±0.001 | C | < 0.01 * | 0.135 |
| 1,4-Benzenediol | 2982.95 | 110 | 0.002±0.00 | 0.109±0.005 | 0.108±0.001 | 0.089±0.006 | 0.095±0.002 | C | 0.743 | 0.025 |
| (<i>Z,Z</i>)-9,12-Octadecadienoic acid (Linoleic acid) | 3177.31 | 81 | 0.154±0.00 | 0.002±0.000 | 0.002±0.000 | 0.003±0.001 | 0.003±0.000 | C | 0.263 | 0.436 |

a: Retention indices (RI) were determined using n-alkanes C11–C32

b: Relative intensity of compounds are displayed as mean ± standard deviation for three independent experiments. Control: control samples inoculated with *Zygosaccharomyces rouxii* and *Tetragenococcus halophilus*. LAB + EtOH: Samples inoculated with *T. halophilus* and added ethanol.

Acid + Yeast: Samples added lactic acid and acetic acid and inoculated with *Z. rouxii*. Acid + EtOH: Sample added lactic acid, acetic acid and ethanol.

c: p-value are calculated by Aloutout 2 software. Significant constituents were marked.

d: Reliability of volatile annotation was shown as follows: A, mass spectrum and retention index were consistent with those of an authentic compound;

B, mass spectrum and retention index were consistent with those of the literature (12, 44-46); C, mass spectrum was consistent with that of the NIST11 MS spectral library.