Design of commercial batch fractionating columns for separation of α- & β-pinenes from turpentine oil by the simple method developed

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A commercial batch fractionating column, for separating α- & β-pinenes from a multicomponent mixture, such as turpentine oil was designed, installed and commissioned. The practical results obtained speak well of the accuracy of the simple graphical method developed for designing such columns and calculations for isolating different components.

The authors developed simple graphical method for designing batch fractionating columns for fractionating multi component mixtures and isolating components in high purity. The method was applied on turpentine oil containing α-pinene, β-pinene and Δ³-carene only. Knowing the number of plates present in the column, it could predict the quantity of high purity α-pinene that could be obtained from a feed of known composition at different reflux ratios. It could also show that if two cuts A and B of same composition are obtained at reflux ratios R1 and R2 respectively, then it is possible to have a single combined cut (A+B) at a reflux ratio R3, such that the time taken for the cuts A and B at R1 and R2 is same as that of (A+B) at R3. It could also predict the composition of the second cut for recovery of β-pinene and its operating reflux.

By applying the method developed, an attempt was therefore made to design a batch column for an industry for fractionating turpentine oil having composition as under, so as to complete a batch of 4000 L in about 24 h and to have maximum cut of 95% pure α-pinene and second cut of β-pinene of maximum possible purity.

Composition of feed turpentine oil

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed %</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>30.0</td>
</tr>
<tr>
<td>β-pinene</td>
<td>10.0</td>
</tr>
<tr>
<td>Δ³-Carene</td>
<td>58.0</td>
</tr>
<tr>
<td>Longifolene</td>
<td>2.0</td>
</tr>
</tbody>
</table>

Design calculations

Taking 100 L of the feed in the reboiler, operating the column at 100 mm Hg Abs. At the top, keeping ΔP at 15±1 mm Hg.

Assuming that the first cut of 20% is obtained of composition

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed %</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>95.0</td>
</tr>
<tr>
<td>β-pinene</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Amount of α- and β-pinene recovered will be

- α-pinene: 20×0.95=19.0
- β-pinene: 20×0.05=1.0

Balance left in the reboiler:

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed %</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>30.0-19.0=11.0</td>
</tr>
<tr>
<td>β-pinene</td>
<td>10.0-1.0=9.0</td>
</tr>
<tr>
<td>Δ³-Carene</td>
<td>60.0-0=60.0</td>
</tr>
</tbody>
</table>

Here longifolene has been taken together with Δ³-Carene, since it is in minute quantity and is high boiling too.

Stream compositions

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed %</th>
<th>Distillate %</th>
<th>Bottom %</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene (LK)</td>
<td>30.0</td>
<td>95.0</td>
<td>13.75</td>
</tr>
<tr>
<td>β-pinene (HK)</td>
<td>10.0</td>
<td>5.0</td>
<td>11.25</td>
</tr>
<tr>
<td>Δ³-Carene (HK+1)</td>
<td>60.0</td>
<td>0.0</td>
<td>75.00</td>
</tr>
</tbody>
</table>

Temperatures: t_vap=90.1°C; t_boe=93.1°C; t_ang=91.6°C
a_LK=1.14; a_HK=1.00; a_HK+1=0.69

Substituting in Fenske’s equation (Fig. 1)

Nm = log [X_LK/X_HK]D (X_HK/X_LK)B / log (a_LK/a_HK)
Nm =log(95/5×11.25/13.75)/ log 1.14 = 20.94

From Fig. 2, Nopt/Nm = 1.7, Nopt = 1.7×20.94 = 35.6
Fig. 1—Fenske equation for minimum plates expressed in graph form

Fig. 2—Relation between optimum-to-minimum ratio and Fenske separation factor of $\alpha_{\text{avg}}$ values

Fig. 3—Optimum-minimum reflux ratio relationship to the column’s feed, distillate, and bottoms composition.

Fig. 4—Underwood’s $\theta$ vs. key ratios in feed

Fig. 5—Relation between reflux ratio and number of plates
Now for Rm/Ropt

\[ \log \left( \frac{X_{LK}}{X_{HK}} \right)_{D} \left( X_{HK}/X_{LK} \right)_{B} \left( X_{LK}/X_{HK} \right)_{F} = 1.49 \]

\[ 0.55 \times 1.14 \]

= log [(15.5454 x (30/10)) = 1.49

From Fig. 3, Ropt/Rm = 1.44

From Underwoods equation for \( \theta \)

\[ \frac{\alpha_{A} X_{DA}/\alpha_{A} + \alpha_{B} X_{DB}/\alpha_{B} - \theta + \alpha_{C} X_{DC}/\alpha_{C} - \theta = 0}{0 = 1.042} \]

To find Rm

\[ \text{Component} \quad \text{Distillate \%} \quad \text{\( \alpha_i \)} \quad \alpha_i/\alpha_i \quad \alpha_i X_{iD}/\alpha_i \]

\[ \begin{array}{llll}
\alpha\text{-pinene} & 95 & 1.14 & 0.08596 \\
\beta\text{-pinene} & 5 & 1.00 & -0.042 \\
\end{array} \]

\[ \text{Rm} + 1 = 9.86, \text{Rm} = 8.86, \text{Ropt} = 8.86 \times 1.44 = 12.76 \]

From Fig. 5

\[ \frac{(n + 1) + (n + 1)m}{(n + 2)} = \frac{(36 - 21.94)}{37} = 0.38 \]

R - Rm/R + 1 = 0.3, R = 13.1

Summary

<table>
<thead>
<tr>
<th>Calculated</th>
<th>Designed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nm = 20.94</td>
<td>Nm = 20.94</td>
</tr>
<tr>
<td>Nopt = 35.6</td>
<td>N = 35.00</td>
</tr>
<tr>
<td>Rm = 8.86</td>
<td>Rm = 8.86</td>
</tr>
<tr>
<td>Ropt = 12.76</td>
<td>R = 13.10</td>
</tr>
</tbody>
</table>

Based on the results of Pilot Plant trial, another cut of about 18% was assumed to take out maximum of \( \alpha\)-, \( \beta\)-pinenes leaving the rest rich in \( \Delta\)\text{-}carene. Therefore, its probable composition assumed is as mentioned in Table 1.

<table>
<thead>
<tr>
<th>Component</th>
<th>( \text{Distillate %} )</th>
<th>( \text{( \alpha_i )} )</th>
<th>( \frac{\alpha_i}{\alpha_i} )</th>
<th>( \frac{\alpha_i X_{iD}}{\alpha_i} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \alpha\text{-pinene} )</td>
<td>95</td>
<td>1.14</td>
<td>0.08596</td>
<td>11.05</td>
</tr>
<tr>
<td>( \beta\text{-pinene} )</td>
<td>5</td>
<td>1.00</td>
<td>-0.042</td>
<td>-1.19</td>
</tr>
</tbody>
</table>

\[ \text{Rm} + 1 = 9.21, \text{Rm} = 8.21, \text{Ropt} = 8.21 \times 1.35 = 11.08 \]

From Fig. 5, \( [(n + 1) + (n + 1)m]/n + 2 = [36 - 19.51]/37 = 0.446 \)

R - Rm/R + 1 = 0.2, R = 10.51

Summary

<table>
<thead>
<tr>
<th>Calculated</th>
<th>Designed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nm = 18.50</td>
<td>Nm = 18.50</td>
</tr>
<tr>
<td>Nopt = 33.32</td>
<td>N = 35.00</td>
</tr>
<tr>
<td>Rm = 8.21</td>
<td>Rm = 8.21</td>
</tr>
<tr>
<td>Ropt = 11.08</td>
<td>R = 10.51</td>
</tr>
</tbody>
</table>

Now let the distillation take 20 h, leaving the rest of 4 h for heating, equilibrium, cooling and discharging the column residue. Let \( X \) L/h be the working boil-up rate of the column.

Time taken for ist cut of 20% (i.e. 4000 x 20/100 = 800 L), at \( R = 13.1:1 \)

Product rate = \( X / 14.1 \) L/h

Time taken = 800 x 14.1/L x h

Time taken for 2nd cut of 18.26% (i.e. 4000 x 18.26/100 = 730.4 L) at \( R = 10.51:1 \)

Product rate = \( X / 11.51 \) L/h

Time taken = 730.4 x 11.51/L x h

Taking the rest 56.74% at \( R = 1:1 \), leaving 5% as residue

Product rate = \( X / 2 \) L/h

Time taken = 4000 x 0.5674 x 2/X

Total time for distillation

\[ 800 \times 14.1/X + 730.4 \times 11.51/X + 4539.2/X = 20 \text{ h} \]

\[ X = 1211.3 \text{ L/h} \]

which is the approximate working boil-up rate. Taking it 75% of flooding rate

\[ \text{Flooding rate} = 1211.3 \times 0.75 = 1615.97 \text{ L/h} \]

Now from Pilot Plant trial

Flooding rate at 100 mm Hg operating pressure for 175 mm dia column = 145.2 L/h

\[ \text{Column dia for flooding rate of 1615.07 L/h} \]
Table 1

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottom</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.49 α-pinene(LK+1)</td>
<td>11.00</td>
<td>13.75</td>
<td>10.20</td>
<td>55.44</td>
<td>0.80</td>
<td>1.30</td>
</tr>
<tr>
<td>1.45 β-pinene(LK)</td>
<td>9.00</td>
<td>11.25</td>
<td>7.80</td>
<td>42.39</td>
<td>1.20</td>
<td>1.95</td>
</tr>
<tr>
<td>1.00 Δ3-carene(HK)</td>
<td>60.00</td>
<td>75.00</td>
<td>0.40</td>
<td>2.17</td>
<td>59.60</td>
<td>96.75</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>80.00</strong></td>
<td><strong>100.00</strong></td>
<td><strong>18.40</strong></td>
<td><strong>100.00</strong></td>
<td><strong>61.00</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

Table 2

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottom</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.49 α-pinene LK-1</td>
<td>11.00</td>
<td>13.75</td>
<td>10.06</td>
<td>55.09</td>
<td>0.94</td>
<td>1.52</td>
</tr>
<tr>
<td>1.45 β-pinene LK</td>
<td>9.00</td>
<td>11.25</td>
<td>7.80</td>
<td>42.72</td>
<td>1.20</td>
<td>1.94</td>
</tr>
<tr>
<td>1.00 Δ3-carene HK</td>
<td>60.00</td>
<td>75.00</td>
<td>0.40</td>
<td>2.19</td>
<td>59.60</td>
<td>96.54</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>75.00</strong></td>
<td><strong>100.00</strong></td>
<td><strong>18.26</strong></td>
<td><strong>100.00</strong></td>
<td><strong>61.74</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

Table 3

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottom</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.14 α-pinene_LK</td>
<td>30</td>
<td>17.64</td>
<td>9.50</td>
<td>12.36</td>
<td>15.18</td>
<td></td>
</tr>
<tr>
<td>1.00 β-pinene_HK</td>
<td>10</td>
<td>0.93</td>
<td>5.0</td>
<td>9.07</td>
<td>11.14</td>
<td></td>
</tr>
<tr>
<td>0.69 Δ3-carene HK+1</td>
<td>60</td>
<td>—</td>
<td>—</td>
<td>60.00</td>
<td>73.68</td>
<td></td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>100</strong></td>
<td><strong>18.57</strong></td>
<td><strong>100.00</strong></td>
<td><strong>81.43</strong></td>
<td><strong>100.00</strong></td>
<td></td>
</tr>
</tbody>
</table>

Table 4

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottom</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.49 α-pinene_LK-1</td>
<td>12.36</td>
<td>15.18</td>
<td>3.0940</td>
<td>65</td>
<td>9.2660</td>
<td>12.09</td>
</tr>
<tr>
<td>1.45 β-pinene_LK</td>
<td>9.07</td>
<td>11.14</td>
<td>1.6184</td>
<td>34</td>
<td>7.4516</td>
<td>9.72</td>
</tr>
<tr>
<td>1.00 Δ3-carene_HK</td>
<td>60.00</td>
<td>73.68</td>
<td>0.0476</td>
<td>1</td>
<td>59.9524</td>
<td>78.16</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>81.43</strong></td>
<td><strong>100.00</strong></td>
<td><strong>4.7600</strong></td>
<td><strong>100.00</strong></td>
<td><strong>76.6700</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

Table 5

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottoms</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>12.36</td>
<td>15.18</td>
<td>3.05</td>
<td>64.65</td>
<td>9.31</td>
<td>12.14</td>
</tr>
<tr>
<td>β-pinene</td>
<td>9.07</td>
<td>11.14</td>
<td>1.62</td>
<td>34.34</td>
<td>7.45</td>
<td>9.71</td>
</tr>
<tr>
<td>Δ3-carene</td>
<td>60.00</td>
<td>73.68</td>
<td>0.0476</td>
<td>1</td>
<td>59.9524</td>
<td>73.15</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>81.43</strong></td>
<td><strong>100.00</strong></td>
<td><strong>4.7176</strong></td>
<td><strong>100.00</strong></td>
<td><strong>76.7120</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

Table 6

<table>
<thead>
<tr>
<th>Component</th>
<th>%</th>
<th>Recoveries</th>
<th>Balance</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>52</td>
<td>11.43×0.52=5.944</td>
<td>9.2660−5.944=3.322</td>
</tr>
<tr>
<td>β-pinene</td>
<td>38</td>
<td>11.43×0.38=4.334</td>
<td>7.4516−4.334=3.109</td>
</tr>
<tr>
<td>Δ3-carene</td>
<td>10</td>
<td>11.43×0.10=1.143</td>
<td>59.9524−1.143=58.809</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>11.430</strong></td>
<td><strong>76.67−1143 65.240</strong></td>
<td></td>
</tr>
</tbody>
</table>

Table 7

<table>
<thead>
<tr>
<th>Component</th>
<th>Feed</th>
<th>%</th>
<th>Distillate</th>
<th>%</th>
<th>Bottom</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>9.266</td>
<td>12.09</td>
<td>5.944</td>
<td>52</td>
<td>3.322</td>
<td>5.09</td>
</tr>
<tr>
<td>β-pinene</td>
<td>7.4516</td>
<td>9.72</td>
<td>4.343</td>
<td>38</td>
<td>7.45</td>
<td>4.77</td>
</tr>
<tr>
<td>Δ3-carene</td>
<td>59.9524</td>
<td>78.19</td>
<td>1.143</td>
<td>10</td>
<td>59.9524</td>
<td>90.14</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>76.6700</strong></td>
<td><strong>100.00</strong></td>
<td><strong>11.430</strong></td>
<td><strong>100.00</strong></td>
<td><strong>65.240</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>
Component  | Feed  | % | Distillate | % | Bottoms  | %
--- | --- | --- | --- | --- | --- | ---
α-pinene  | 9.2660 | 12.09 | 6.078 | 52.56 | 3.188 | 4.90
β-pinene  | 7.4516 | 9.72 | 4.343 | 37.56 | 3.109 | 4.77
Δ³-carene | 59.9524 | 78.19 | 1.143 | 9.88 | 58.809 | 90.33

Table 8

Component  | Feed  | % | Distillate | % | Bottoms  | %
--- | --- | --- | --- | --- | --- | ---
α-pinene  | 12.36 | 15.18 | 11.35 | 57.76 | 1.01 | 1.64
β-pinene  | 9.07  | 11.14 | 7.90  | 40.20 | 1.17 | 1.89
Δ³-carene | 60.00 | 73.68 | 0.40  | 2.04  | 59.60 | 96.47

Table 9

<table>
<thead>
<tr>
<th>Cut</th>
<th>No.</th>
<th>% of feed</th>
<th>vol. lit</th>
<th>Reflux Ratio</th>
<th>Composition %</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>1</td>
<td>20.00</td>
<td>20.00</td>
<td>13.10</td>
<td>α-p 95  β-p 5 Δ³-c -</td>
</tr>
<tr>
<td>II</td>
<td>3</td>
<td>18.26</td>
<td>18.26</td>
<td>10.51</td>
<td>α-p 42.72  β-p 2.19 Δ³-c</td>
</tr>
<tr>
<td>III</td>
<td>4</td>
<td>61.74</td>
<td>61.74</td>
<td>-</td>
<td>α-p 93.30  β-p 3.24 Δ³-c</td>
</tr>
</tbody>
</table>

Table 10

<table>
<thead>
<tr>
<th>Cut</th>
<th>No.</th>
<th>% of feed</th>
<th>vol. lit</th>
<th>Reflux Ratio</th>
<th>Composition %</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>1</td>
<td>18.57</td>
<td>780</td>
<td>14.65</td>
<td>α-p 95  β-p 5 Δ³-c -</td>
</tr>
<tr>
<td>II</td>
<td>2</td>
<td>4.76</td>
<td>200</td>
<td>9.24</td>
<td>α-p 65  β-p 4  Δ³-c -</td>
</tr>
<tr>
<td>III</td>
<td>3</td>
<td>11.43</td>
<td>480</td>
<td>8.56</td>
<td>α-p 52  β-p 3  Δ³-c -</td>
</tr>
<tr>
<td>IV</td>
<td>4</td>
<td>47.38</td>
<td>1990</td>
<td>-</td>
<td>α-p 5.09  β-p 4.77 Δ³-c 3.07</td>
</tr>
<tr>
<td>Residue</td>
<td>5</td>
<td>14.29</td>
<td>600</td>
<td>-</td>
<td>α-p 87.07  β-p 3.07 Δ³-c 3.07</td>
</tr>
<tr>
<td>Losses</td>
<td>6</td>
<td>3.57</td>
<td>150</td>
<td>-</td>
<td>α-p 93.23  β-p 1.89 Δ³-c 3.24</td>
</tr>
</tbody>
</table>

Table 11

D²/[175 x 175] = 1615.07/145.2, D = 583.65 mm
Rounding up gives a column dia of 600 mm
:: Flooding rate for 600 mm dia = 1615.07 x 600 x 600/[583.65 x 583.65] = 1708.8 L/h

Checking column dia through basic calculations

The molecular weight of the turpentine components ie. α-pinene, β-pinene and Δ³-carene, are the same = 136. Now volume of one gram mole of a component at NTP = 22.4 L or for 136 kg oil = 22.4 M³
Now the boiling point of α-pinene at 100 mm Hg = 90.1°C

273 + 90.1 = 363.1°C

From Gas Law, (760 x 22.4)/273 = (100 x V2)/363.1
V2 = 226.42544 M³ for 136 Kg α-pinene
Sp. Gravity of α-pinene = 0.86
Vol. of 136 Kg = 136/0.86 = 158.14 L
or volume of 136 Kg or 158.14 L of liquid α-pinene at 100 mm Hg at 90.1°C = 226425.44 L
:: Vapour density = 0.0006 g/cc or Kg/L
Vapour volume for 1000 L α-pinene = 226425.44 x 1000/158.14 = 1431803.7 L
If 1000 L of liquid α-pinene be the boil-up rate of the column per h,
its vapour volume/s = 1431803.7/3600 = 397.723 L/s
\[ \times 0.03532 = 14.047 \text{ cft/s} \]

Now vapour velocity is given by:
\[ V_s \text{ al} = 0.137 \left[ \frac{(P_L - P_v)}{P_v} \right]^{0.57} = 0.137 \left[ \frac{(0.86 - 0.0006)}{0.0006} \right]^{0.57} = 8.623 \text{ ft/s} \]

Crosssection area of the column = 14.047/8.623 = 1.646 ft²

Now voidage of steel Pall rings = 94% also taking 75% as the working boil up rate
\[ \therefore \text{Cross section area} = \frac{14.047}{8.623 \times 0.94 \times 0.75} = \pi D^2 / 4 \]

Column dia D = 1.7152 ft × 12 × 25.4 = 522.8 mm
\therefore Working boil-up rate for 600 mm dia column = (1000 × 600 × 600)/522.8 × 522.8 = 1317 L/h
\therefore Column flooding rate = 1317/0.75 = 1756 L/h (Actual column flooding rate obtained = 1700 L/h which matches well).

Number of plates in the commercial packed column designed

The column designed (Fig. 6) had the following features:

Column dia = 600 mm
No. of sections in the column = 5
Total height of each section = 2700 mm
Packed height of first 4 sections = 2300 mm each
Packed height of the top section = 2500 mm

Each section of the column was provided with a distributor at the top, above the packing. The first 4 distributors were of 300 mm height and the top one of 100 mm. The first 4 sections were made of M.S. and the top section of S.S.-304. The lowest section was packed with 38 mm M.S. Pall rings, the middle three sections with 25 mm M.S. Pall rings and the top section with 25 mm S.S. 304 Pall rings.

The number of plates were calculated as under:

\[ \text{H.E.T.P.} = K_1 G^{K_2} d^{K_3} h^{K_4} \frac{\alpha \mu}{\rho} \]

where \(K_1, K_2, K_3\) are constants and functions of the packing. Their values for steel rasching rings are:

(i) for 25 mm rings, \(K_1 = 0.57, K_2 = -0.10, K_3 = 1.24\)
(ii) for 50 mm rings, \(K_1 = 0.42, K_2 = 0, K_3 = 1.24\)

\[ G = \text{Superficial mass velocity of vapour 1b/hr (sq.ft)} \]
\[ d = \text{column diameter, inches} \]
\[ h = \text{height of packing, ft} \]
\[ \alpha = \text{relative volatility} = 1.14 \]
\[ \mu = \text{liquid viscosity centipoise} = 0.55 \]
\[ \rho = \text{liquid density, g/c.c.} = 0.86 \]

\[ \text{H.E.T.P.} = \text{Height of packing equivalent to one theoretical plate, inches} \]

Substitution of values gives:

\[ \text{Column dia} = 600/25.4 \times 12 = 1.9685 \text{ ft} \]
\[ G = (1700 \times 0.86 \times 2.2 \times 4 \times 0.75)/\pi (1.9685) = 792.63 \]

For 25 mm Rasching rings
\[ G^{K_2} = (792.63)^{-0.10} = 0.51297 \]
\[ d^{K_3} = (600/25.4)^{1.24} = 50.456 \]
\[ h^{K_4} = (2300/25.4 \times 12)^{1.96} = 1.96 \]

also for top section of packed height of 2500 mm
\[ h^{K_4} = (2500/25.4 \times 12)^{1.96} = 2.015 \]

For 50 mm Rasching rings
\[ G^{K_2} = (792.63)^{-1} \]
\[ d^{K_3} = (600/25.4)^{1.24} = 50.456 \]

From above.

(i) HETP for middle 3 sections packed with 25 mm M.S. Pall rings:
Now HETP for steel Rasching rings = 0.57 × 0.51297 × 50.456 × 1.96 × 1.14 × 0.55/0.86 = 21.08 inches

Now efficiency of steel Rasching rings is around 70% of steel Pall rings.
\[ \therefore \text{HETP for steel Pall rings} = 21.08 \times 0.7 = 14.756 \text{ inches} \]

(ii) HETP for top section packed with 25 mm S.S. Pall rings, Packed height = 2500 mm:
HETP for steel Rasching rings = 0.57 × 0.51297
HETP for steel Pall rings = 21.67x0.7 = 15.169 inches

HETP for 50mm Pall rings = 30.28x0.7 = 21.197 inches

HETP for steel Rasching rings = 0.42 x 1.0 x 50.456 x 1.96 x 1.14 x 0.55 / 0.86 = 29.936

HETP for steel Pall rings = 21.67 x 0.7 = 15.169 inches

HETP for 50mm Pall rings = 30.28 x 0.7 = 21.197 inches

HETP for 38mm Pall rings = (21.197 + 15.169) / 2 = 17.183 inches

HETP for 38mm Pall rings = (21.197 + 15.169) / 2 = 17.183 inches

Now taking the HETP for 38mm Pall rings as average HETP of 25mm and of 50mm, Pall rings, we get

HETP for 38mm Pall rings = (17.183 + 21.197) / 2 = 19.190 inches

Now total no. of plates in the column = (2300 / 25.4 x 0.55) + (2300 x 3 / 25.4 x 14.756) + (2500 / 25.4 x 15.169) = 5.037 + 18.410 + 6.489 = 30.036

Now total no. of plates = 30 + 1 = 31

The above column was fabricated, installed and commissioned under the guidance of the author.

Results and Discussions

**Practical results obtained from the above column**

Composition of turpentine charged

<table>
<thead>
<tr>
<th>Component</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene</td>
<td>30%</td>
</tr>
<tr>
<td>β-pinene</td>
<td>10%</td>
</tr>
<tr>
<td>Δ^3-carene</td>
<td>58%</td>
</tr>
<tr>
<td>Longifolene</td>
<td>2%</td>
</tr>
</tbody>
</table>

Batch charged in the reboiler = 4200 L

Flooding rate of the column at 100 mm Hg at the top of the column = 1700 L/h

Column AP (working) = 30±2 mm Hg

The average values of α, which do not change materially are:

α_LK = 1.14, α_HK = 1.0, α_HK+1 = 0.69

Product cut at R = 14:1

1st cut (18.57% of charge) = 780 L

α-pinene = 95%

β-pinene = 5%

Product cuts at R = 10:1

2nd cut (4.76% of charge) = 200 L

α-pinene = 65%

β-pinene = 34%

Δ^3-carene = 1%

3rd cut (11.43% of charge) = 480 L

α-pinene = 52%

β-pinene = 38%

Δ^3-carene = 10%

**IVth cut 47.38% of charge**

at no reflux, called D.D. Turpentine = 1990 L

Residue (14.29% of charge)

called Pine tar = 600 L

Losses (3.57% of charge) = 150 L

Total 4200 L

Analysis of the results by applying simple graphical method developed

For a charge of 100 L

Considering 1st cut of 18.57%, containing:

α-pinene 95%

β-pinene 5%

Component | Recoveries made | Balance left

<table>
<thead>
<tr>
<th>Component</th>
<th>Distillate %</th>
<th>α</th>
<th>α-θ/α</th>
<th>αθθ/α-θ</th>
</tr>
</thead>
<tbody>
<tr>
<td>a-pinene</td>
<td>95</td>
<td>1.14</td>
<td>0.086</td>
<td>11.051</td>
</tr>
<tr>
<td>β-pinene</td>
<td>5</td>
<td>1.00</td>
<td>-0.042</td>
<td>-1.190</td>
</tr>
<tr>
<td>Δ^3-carene</td>
<td>0.69</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Rm + 1 = 9.86, Rm = 8.86, From Fig. 5

[(n + 1) - (n + 1)m]/n + 2 = (32-21)/33 = (0.33R - Rm)/R + 1 = 0.37, R = 14.65

Summary

<table>
<thead>
<tr>
<th>Calculated</th>
<th>Designed</th>
<th>Actual R used</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nm = 20.11</td>
<td>Nm = 20.11</td>
<td>R = 14:1</td>
</tr>
<tr>
<td>Nopt = 35.6</td>
<td>Nopt = 35.6</td>
<td>R = 14:1</td>
</tr>
<tr>
<td>Rm = 8.86</td>
<td>Rm = 8.86</td>
<td>R = 14.65</td>
</tr>
<tr>
<td>Ropt = 12.76</td>
<td>Ropt = 12.76</td>
<td>R = 14.65</td>
</tr>
</tbody>
</table>

From the column the distillate was collected at the rate of 85 to 90 L/h

Now column flooding rate = 1700 L/h, then distillate at

80% of Flooding rate = 1360 L/h = 90.67 L/h
75% of Flooding rate = 1275 L/h + 15 = 85.0 L/h
70% of Flooding rate = 1190 L/h + 15 = 79.33 L/h
This shows that the product was taken at R = 14:1 and product was collected by operating the column at 75% to 80% of flooding rate.

Considering the calculated flooding rate of 1756 L/h, then distillate at 

80% of flooding rate = 404.8 L/h + 15 = 93.65 L/h
80% of flooding rate = 404.8 L/h + 15 = 89.37 L/h

75% of flooding rate = 1317 L/h + 15 = 87.8 L/h
75% of flooding rate = 1317 L/h + 15 = 84.15 L/h

Hence, a reflux ratio of R = 14.65 is not ruled out for 75% to 80% of flooding rate.

IInd cut of 4.76% at R = 10:1

Composition of the cut

<table>
<thead>
<tr>
<th>Component</th>
<th>Distillate %</th>
<th>α</th>
<th>α-0/α-1</th>
<th>α×d/α-0</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene 5</td>
<td>65</td>
<td>1.49</td>
<td>0.1208</td>
<td>5.3806</td>
</tr>
<tr>
<td>β-pinene 3</td>
<td>34</td>
<td>1.45</td>
<td>0.0965</td>
<td>3.5233</td>
</tr>
<tr>
<td>Δ3-carene 1</td>
<td>1</td>
<td>1.00</td>
<td>-0.31</td>
<td>-0.0323</td>
</tr>
</tbody>
</table>

Rm = 7.87, Ropt = 7.87 × 1.32 = 10.39

From Fig. 5, [(n + 1) - (n + 1)m]/n + 2 = (32 - 16.1)/33 = 0.48
R = Rm/R + 1 = 0.17, R = 9.24

Summary

Calculated  Designed
Nm = 15.10  Nm = 15.10
Nopt = 28.50  N = 31.00
Rm = 7.87  Rm = 7.87
Ropt = 10.39  R = 9.24

Checking the distribution of Non key components

αmean = (αlakav + αhikav)/2 = (1.45 + 1.0)/2 = 1.225

For the light key component α-pinene (di > bi)

αα-pinene > αmean

Reference component r is the heavy key Δ3-carene

bi = fi/[1 + (di/bi)αav/αav] and di = fi - bi

For α-pinene

bi = 12.36/[1 + (0.0476/59.9524) (1.49/1.00) 15.1] = 9.3123

New compositions are shown in Table 5.

Hence, the composition and cut percentage is almost matching Cut III of 11.43% at R = 10:1.

Composition of the cut and recoveries made are given in Table 6 and new compositions are shown in Table 7.

From Fenske’s equation

Nm = log (38 × 90.14/10 × 4.77)/log 1.45 = 11.5

From Fig. 2, Nopt/Nm = 1.99, Nopt = 11.5 × 1.99 = 22.89

For Ropt/Rm, log [71.803 × (9.72/78.19) 0.55 × 1.45] = log 13.165 = 1.134

From Fig. 3, Ropt/Rm = 1.29

From Underwoods equation for 0, 0 = 1.33, to find Rm,

<table>
<thead>
<tr>
<th>Component</th>
<th>Distillate %</th>
<th>α</th>
<th>α-0/α-1</th>
<th>α×d/α-0</th>
</tr>
</thead>
<tbody>
<tr>
<td>α-pinene 5</td>
<td>52</td>
<td>1.49</td>
<td>0.1074</td>
<td>4.8417</td>
</tr>
<tr>
<td>β-pinene 3</td>
<td>38</td>
<td>1.45</td>
<td>0.0827</td>
<td>4.9949</td>
</tr>
<tr>
<td>Δ3-carene 10</td>
<td>10</td>
<td>1.00</td>
<td>-0.33</td>
<td>-0.3030</td>
</tr>
</tbody>
</table>

Rm = 8.13, Ropt = 8.13 × 129 = 10.49
From Fig. 5, \[\frac{(n+1)-(n+1)m}{n+2} = \frac{32-12.5}{33} = 0.376\]
\[R - R_m/R + 1 = 0.33, R = 8.56\]

**Summary**

Calculated  
Designed

\[Nm = 11.50 \quad Nm = 11.50\]
\[N_{opt} = 22.89 \quad N = 31.00\]
\[R_m = 8.13 \quad R_m = 8.13\]
\[R_{opt} = 10.49 \quad R = 8.56\]

Checking the distribution of Non key component, \(\alpha\)-pinene

\[b_i = 9.266/[1 + (1.143/58.809) (1.49/1.00)^{11.5}] = 3.188\]
\[d_i = f_i - b_i = 9.266 - 3.188 = 6.078\]

New compositions will be as mentioned in Table 8. Here also the composition and cut percentage is almost matching.

**Conclusions**

The column was operated at \(R = 14:1\), during 1st cut and
\(R = 10:1\) during 2nd and 3rd cuts.
It was operated at no reflux for IVth cut.

*From the analysis by simple graphical method applied*

(i) For the 1st cut of 18.57%  
The composition, cut percentage as well as reflux ratio matches well. Here the purity of distillate is 95%, the minimum prescribed in the method.

(ii) For cuts II and III  
Here the column was operated at \(R = 10:1\), where as the calculations show that the column should have been operated at \(R = 9.24:1\) for cut II and \(R = 8.56\) for cut III. This difference could be attributed to the fact that none of the key components either in the distillate or in the bottom of cuts II & III had the purity 95%, the primary conditions laid down in the method.

(iii) In case of cut II  
The method readily indicated that the selection of \(\alpha\)-pinene and \(\beta\)-pinene as the key components was wrong. It had to be \(\beta\)-pinene and \(\Delta^3\)-carene only.

**Special Case**

Considering that in place of cuts II and III, a single cut is taken, so as to recover most of \(\alpha,\beta\)-pinenes in the distillation, along with small amount of \(\Delta^3\)-carene, the heavy key component. The amount of \(\Delta^3\)-carene should be \(\leq 5\%\) of \(\beta\)-pinene, the light key component. Similarly leaving small amount of \(\beta\)-pinene in the bottoms. It should also be \(\leq 5\%\) of \(\Delta^3\)-carene the heavy key component, so that the method developed is applicable.

Composition of streams assumed is given in Table 9.
From Fenske's equation
\[Nm = \log \left(40.20 \times 96.47/2.04 \times 1.89\right)/\log 1.45 = 18.6\]
Checking the distribution of Non key component, \(\alpha\)-pinene,
\[b_i = 12.36/[1 + (0.40/59.60) (1.49/1.00)^{18.6}] = 1.015\]
\[d_i = f_i - b_i = 12.36 - 1.015 = 11.345\]
Hence the values chosen match very well with the calculated values.

From Fig. 2, \(N_{opt}/Nm = 1.8, N_{opt} = 18.6 \times 1.8 = 33.48\)
For \(R_{opt}/R_m, \log [1005.83 \times (11.14/73.68)(0.55 \times 1.45)] = 2.348\)
From Fig. 3, \(R_{opt}/R_m = 1.355\)
From Underwoods equation for \(\theta, \theta = 1.31\), to find \(R_m\)

\[
\text{Compo-} \quad \text{Distillate\%} \quad \alpha_i \quad \alpha_{i-0}/\alpha_i \quad \alpha_{i-xd}/\alpha_{i-0}
\]
\[
\alpha\text{-pinene} \quad 57.76 \quad 1.49 \quad 0.1208 \quad 4.7815
\]
\[
\beta\text{-pinene} \quad 40.20 \quad 1.45 \quad 0.0965 \quad 4.1658
\]
\[
\Delta^3\text{-carene} \quad 2.04 \quad 1.00 \quad -0.31 \quad -0.0658
\]
\[
R_m = 7.88, R_{opt} = 7.88 \times 1.335 = 10.68
\]
From Fig. 5, \([n+1)-(n+1)m]/n+2 = 19.63/33 = 0.376\)
\[R = R_m/R + 1 = 0.33, R = 11.69\]

**Summary**

Calculated  
Designed

\[Nm = 18.60 \quad Nm = 18.60\]
\[N_{opt} = 33.48 \quad N = 31.00\]
\[R_m = 7.88 \quad R_m = 7.88\]
\[R_{opt} = 10.68 \quad RR = 11.69\]

Hence the recoveries could have been best made by taking a single cut of 19.65% at \(R = 11.69:1\).

**Summary of Results**

<table>
<thead>
<tr>
<th>Feed Composition</th>
<th>(\alpha)-pinene</th>
<th>30%</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\beta)-pinene</td>
<td>10%</td>
<td></td>
</tr>
<tr>
<td>(\Delta^3)-carene</td>
<td>58%</td>
<td></td>
</tr>
<tr>
<td>Longifolene</td>
<td>02%</td>
<td></td>
</tr>
</tbody>
</table>
(i) For column with 35 theoretical plates and Feed = 100 L. Average composition of various cuts is mentioned in Table 10.
(ii) From commercial column having theoretical plates 31, Feed = 4200 L. Average composition of various cuts is given in Table 11. Recommended operation is mentioned in Table 12.

Conclusions
The method tried on practical results obtained from commercial column has proved to be quite accurate within the limits of \( \geq 95\% \) purity of the light key component with respect to heavy key component in the distillate. It has also proved that it can predict, in case the choice of light and heavy key components or the volume of the cut or its composition chosen is wrong. In such case the values calculated for Figs 1, 2, 3 and 4 shall not fit in the graphs.

References