TECHNO-ECONOMIC EVALUATION OF RESIDUE EXHAUSTION IN BATCH RECTIFICATION ETHANOL PRODUCTION PLANT

by

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This paper presents the techno-economic optimization of batch plant for production of rectified alcohol based on the concentration of ethanol in residue. The aim of the analysis was to determine the extent to which it is economically profitable to exhaust the residual liquid in boiler. The "profit production" criterion is used for calculations.

Key words: *rectified ethanol, plant optimization, biofuels, case study*

Introduction

When it comes to ethanol production, rectification is the final operation in which the obtained distillate has ethanol volume fraction 96.2 vol.% Rectification plants can work as continuous (characterized by stationary operating regime) or batch (with non-stationary operating regime). The advantage of batch distillation is that the distillate has higher quality (lower fraction of impurities) and a basic shortage is the higher production cost. The most influential factors in bioethanol productions and its impact on the production cost was presented in [1].

Batch rectification plant, analyzed in this paper, is built in Serbia (vilage Kostojevići) and has a nominal production capacity of $\dot{V}_{\mathrm{D,AA,nom}}$ = 4000 LAA per day [2]. First phase of distillation is the continuous production of raw (unrectified) alcohol with 88 vol.% of ethanol (0.658208 kmol/kmol) and the specific content of impurities [2]. In second phase of production raw ethanol is rectified (refined) in batch distillation plant to meet the requirements of relevant standards for rectified alcohol. The final product (distillate) contains 96.2 vol.% of ethanol. In addition, distillate contains water and small amounts of impurities such as aldehydes, methanol, esters, fusel oils, acids, *etc*.

Performance of the batch rectification ethanol production plant is defined by several factors usually defined in the plant operating manual: the feed preparation procedure in the reboiler (pot), the amounts of the fractions (head, heart and feints), the concentration of ethanol in the individual fractions and so on. For specific industrial facility the operating procedure is given in the next section.

During the batch rectification ethanol concentration in reboiler continuously decreases. After a certain moment it is no longer possible to obtain the distillate with desired ethanol concentration (96.2 vol.%), and tail (end) fractions are characterized with lesser ethanol con-

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tent. Having in mind that the ethanol content in end fractions is still significant (on average about 90 vol.%), those fractions can be further processed in the next batch.

In open literature, there are some indications for the limit of the residue exhaustion profitability. For example, for the continuous distillation columns the recommended ethanol limit in residue is 39-124 ppm_{mol}, but in [2] it was shown that the optimal solution is much higher (250-300 ppm_{mol}).

In batch rectification plant, the final separation of fractions is followed with continually increase of the reflux in order to produce the specified distillate composition. Greater values of reflux increase the production cost of the distillate and according to [3] batch rectification process should not be lead with the reflux greater than 15-30, if the economic production is the goal.

The aim of hereby presented analysis is to determine the extent to which the economically profitable residue exhaustion is obtained.

Description of the industrial plant for batch rectification

Simplified process flow diagram for rectified ethanol production plant is shown in fig. 1. The plant works in cycles, whereby each cycle operates with three batches. The raw material for rectification plant is raw ethanol obtained from continuous distillation column. It is mixed with water (the final content of ethanol is around 60 vol.%) to ensure effective removal of impurities (methanol, aldehydes, esters, *etc*).

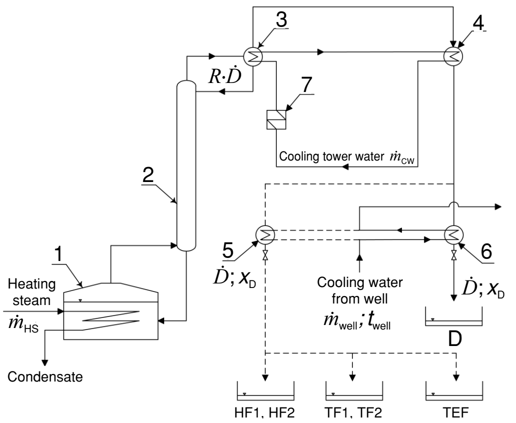

Figure 1. Flow diagram of batch rectification plant
1 – reboiler, 2 – rectification column, 3 – dephlegmator, 4 – condenser, 5 – heat exchanger, 6 – heat exchanger, 7 – cooling tower

The raw material in reboiler (1) is heated by saturated water steam (p_{steam} = 6 bar), then it evaporates, the distillate vapor leaves rectification column (2) and goes to dephlegmator (3). A liquefied distillate from dephlegmator returns at the top of the column as a reflux. The rest of the distillate vapor goes to condenser (4). Cold fluid for dephlegmator and condenser is water from cooling tower (7). The cooling tower outlet water temperature is 28 °C (corresponds with air wet bulb temperature is t_{wb} = 24 °C). After condensation distillate is cooled in heat exchanger (5) or (6), and then stored in the appropriate tanks. For additional distillate cooling in (5) or (6) the water from the local well is used (t_{well} = 15 °C, flow rate $\dot{m}_{well}$).

Before the final product the head fractions, HF1 and HF2, are separated. Beside ethanol, those fractions also contain significant amount of more volatile components like methanol, aldehydes and esters, so that is why the fractions HF1 and HF2 are removed from process. From the total amount of ethanol in the reboiler before starting process, about 8.5% (710 LAA) is removed with fractions HF1 and HF2. After HF1 and HF2 the final product (heart fraction) is separated. The final product of the rectification plant is the distillate with ethanol content of 96.2 vol.% = 94.2 %mas. From the total amount of ethanol in the reboiler at the start of the process, about 84% (7020 LAA) is removed with fraction D. Described algorithm is carried out in all three batches, but at the end of the third batch the tail fractions, TF1 and TF2, are separated. The ethanol content in the tail fractions TF1 and TF2 are 94 vol.% and 85 vol.% , respectively. Tail fractions also contain significant amount of fusel alcohols (fusel oils = higher-order alcohols), acids, esters and less volatile fractions. The TF1 and TF2 are used in the next rectification cycle. From the total amount of ethanol in the reboiler before starting process, about 4.8% (400 lAA) removed with tail fractions. After TF1 and TF2 separation, there is a small amount of ethanol in the reboiler (2.7% from starting value or 225 LAA). The process of distillation can be further unrolling in a process called exhaustion. The ethanol content in the distillate during exhaustion is 85 %vol.

Rectification process description was carried out using technical documentation of distillation plant [4] which is based on the instruction [5] and the recommendations presented in [6]. The rectified ethanol (heart fraction) production rate was based on absolute alcohol flow rate of $\dot{V}_{\mathrm{D,AA,nom}}$ = 4000 LAA per day = 166.67 LAA per hour.

Economic analysis of rectified ethanol production

Rectified ethanol production overall production costs (C_{tot}, € per year) are calculated as the sum of investment or capital costs (C_{inv}, €) and operating costs (C_{op}, € per year) [7]:

$$C_{\mathrm{tot}} = aC_{\mathrm{inv}} + C_{\mathrm{op}} \tag{1}$$

Service life of this kind of production plant is at least ten years, but can be as long as 30 years [7], so we used three amortization rates: a = 1/10 per year, a = 1/20 per year and a = 1/30 per year for 10, 20, and 30 years of service life.

Investment (capital) cost

Estimation of investment costs (C_{inv}, €) was done by using detailed factorial method [8] in which the plant investment costs are estimated as a function of capital cost of all the major equipment items or major process units (C_{BE}, €). The following equation was used:

$$C_{\mathrm{inv}} = C_{\mathrm{BE}}\left(1+\sum_{i=1}^{9} f_i\right)\left(1+\sum_{i=10}^{13} f_i\right) \tag{2}$$

and typical values for direct and indirect cost factors f_i were adopted from [7].

Major equipment costs were obtained using procedures from [9-11], and included the units presented in fig. 1: reboiler (1), rectification column (2), dephlegmator (3), condenser (4), two distillate coolers (5) and (6), and water cooling tower (7).

Considering the composition of working fluids and desired quality of distillate, stainless steel was the material chosen for elements in contact with process fluids, while the carbon steel is chosen for other parts of equipment.

Reboiler is a batch heat exchanger with helical tube coils. Total volume of reboiler is 18 m^3, and its heat surface is 19.6 m^2. Shell and helical tube are made of stainless steel. The reboiler price is estimated using prices for materials.

The cost of rectification column (C_{Col}, €) is determined by the expression [9]:

$$C_{\mathrm{Col}} = C_{\mathrm{SC}} + N_{\mathrm{R}} \cdot C_{\mathrm{T}} \tag{3}$$

where the cost of column shell (C_{SC}) depends on shell diameter, height, material and pressure, and cost of each tray (C_{T}) depends on column diameter, tray type and material. Column diameter is D_{C} = 0.55 m and it was determined by adopting the flood factor FF = 70%. The actual number of trays was determined according to methodology described in [2] and [10], and it is N_{R} = 51. Column shell and trays are made of stainless steel.

Dephlegmator and condenser are the shell-and-tube heat exchangers with fixed tube sheets, and their cost (C_{HE}, €) are estimated as a function of heat transfer surface using the equation from [9]:

$$C_{\mathrm{HE}} = C_{\mathrm{HE,B}} F_{\mathrm{t}} F_{\mathrm{M}} F_{\mathrm{P}} \tag{4}$$

Table 1. Equipment prices in €

No.	Equipment	Price
1	Reboiler	24390
2	Rectification column	70465
3	Dephlegmator	54190
4	Condenser	14450
5	Distillate cooler	9560
6	Distillate cooler	13110
7	Cooling tower	4270
–	Total (C_{BE})	190435

Heat transfer surface of dephlegmator is 56.4 m^2, and for condenser it is 15.6 m^2.

Heat exchangers for final product (5) and for all other fractions (6) are stainless steel tubular exchangers with helical coils having heat transfer surface of 5.69 m^2 each.

Cooling tower cost is estimated on the basis of water flow rate according to [11].

Calculated prices for all equipment are shown in tab. 1.

According to eq. (1) the total investment costs for rectification plant was determined:

$$C_{\mathrm{inv}} = C_{\mathrm{BE}} \left(1 + \sum_{i=1}^{9} f_i\right)\left(1 + \sum_{i=10}^{13} f_i\right) = 190435(1+2.4)(1+0.45) = 938845 \text{ EUR}$$

Operating costs

Steam production operating costs mainly depend on the type of the fuel. We have analyzed natural gas, fuel oil, and coal as three most commonly used fuels and the 6 bar steam production cost are presented in tab. 2. The costs of other utilities are [7]: cooling tower water 0.0067 € per m^3, well water 0.05 € per m^3. Raw material for a rectification process is raw ethanol obtained in continuous distillation plant. The production cost of raw ethanol with detailed explanations is given in [2].

Table 2. Steam production costs [2]

Fuel type	Natural gas	Heavy fuel oil	Coal
Lower heating value	33.34 MJmN^{-3}	40 MJkg	18.2 MJkg^{-1}
Local fuel price	0.458 € per mN^3	390.5 € per tone	100 € per tone
Boiler efficiency, [%]	90	90	80
Steam cost, € per tone	36.06	24.46	15.51

Consideration of the economic profitability of exhausting

After tail fractions (TF1 and TF2) removing, the process of distillation can be further unroll in a process called exhaustion when the tail exhausting fraction (TEF) is obtained. The ethanol content in TEF is 85 vol.% In order to get required ethanol content in the distillate, it is necessary to constantly increase the reflux ratio, because the ethanol content in the reboiler constantly decreases. That is why the distillate flow rate ($\dot{V}_{\mathrm{D}}$, 1 per hour) will constantly decrease leading to decrease in amount of produced ethanol ($\dot{V}_{\mathrm{D,AA}}$, LAA per hour) and to increase of production cost reduced to 1 LAA (€ per LAA). Taking this into consideration, it is crucial to obtain economically justified period of production.

During the exhaustion of the reboiler content the main production costs are connected to steam and cooling water. Consideration of the economic profitability of exhausting was conducted using the *profit production criterion* according to inequality. A condition of the economic profitability of exhaustion is the criterion named *profit production* that is based on inequality:

$$C_{\mathrm{EP}} > \frac{aC_{\mathrm{inv}}}{V_{\mathrm{D,AA,year}}} + \left[s_{\mathrm{HS}}(\tau) + s_{\mathrm{HS,av}}\right] C_{\mathrm{HS}} + \left[s_{\mathrm{CW}}(\tau) + s_{\mathrm{CW,av}}\right] C_{\mathrm{CW}} + \left[s_{\mathrm{well}}(\tau) + s_{\mathrm{well,av}}\right] C_{\mathrm{well}} + \left(C_{\mathrm{tot}}^{\mathrm{AA}}\right)_{\mathrm{I}} \tag{5}$$

The market price of ethanol includes the production costs but also the profit of the manufacturer. In further analysis we used the market price C_{EP} = 0.93 € per LAA given to us by the manufacturer. This price is slightly higher then the prices on the European market; for example in Italy price is 0.88 € per LAA, and in Germany it is 0.82 € per LAA.

The consumption of the steam and water (from cooling tower and from well) were determined from material and energy balance for the whole period of exhaustion. Those values are shown in tab. 3.

Based on these data, the specific consumption of steam and water are given in tab. 4.

Table 3. Consumption of steam and water during exhaustion

τ	$x_W(\tau)$ kmol/kmol	$R(\tau)$	$\varepsilon_{D,TEF}$ LAA per liter	$\dot{V}_D(\tau)$ l per hour	$\dot{V}_{DAA}(\tau)$ l per hour	$\dot{m}_{HS}(\tau)$ kg per hour	$\dot{V}_{CW}(\tau)$ m^3 per hour	$\dot{V}_{Well}(\tau)$ m^3 per hour
0.0	0.00913	5.68	0.85	96.1	81.7	284.0	4.2	0.6179
1.0	0.00656	7.89	0.85	69.7	59.2	270.0	4.0	0.4475
2.0	0.00470	11.00	0.85	50.1	42.6	260.0	3.9	0.3202
3.0	0.00336	15.39	0.85	35.8	30.4	251.0	3.8	0.2306
4.0	0.00239	21.55	0.85	25.6	21.8	246.0	3.7	0.1652
5.0	0.00170	30.25	0.85	18.3	15.6	243.0	3.7	0.1170
6.0	0.00121	42.54	0.85	13.0	11.1	240.0	3.7	0.0843
7.0	0.00086	59.89	0.85	9.3	7.9	238.0	3.6	0.0602
8.0	0.00061	84.38	0.85	6.6	5.6	236.0	3.6	0.0430
9.0	0.00043	118.81	0.85	4.6	3.9	234.0	3.6	0.0293
10.0	0.00031	167.26	0.85	3.3	2.8	233.0	3.6	0.0207
11.0	0.00022	235.56	0.85	2.3	2.0	233.0	3.6	0.0155
12.0	0.00015	331.93	0.85	1.7	1.4	233.0	3.6	0.0103
13.0	0.00011	467.92	0.85	1.2	1.0	232.0	3.5	0.0069
14.0	0.00008	659.90	0.85	0.8	0.7	232.0	3.5	0.0052
15.0	0.00005	931.03	0.85	0.6	0.5	232.0	3.5	0.0034
15.9	0.00004	1269.53	0.85	0.4	0.3	232.0	3.5	0.0034

Table 4. Specific consumption of steam and water during exhaustion

τ	$x_W(\tau)$ kmol/kmol	$R(\tau)$	$s_{HS}(\tau)$ kg per LAA	$s_{CW}(\tau)$ m^3 per LAA	$s_{Well}(\tau)$ m^3 per LAA
0.0	0.00913	5.68	3.48	0.0514	0.00758
1.0	0.00656	7.89	4.56	0.0675	0.00758
2.0	0.00470	11.00	6.11	0.0916	0.00758
3.0	0.00336	15.39	8.25	0.1249	0.00758
4.0	0.00239	21.55	11.31	0.1700	0.00758
5.0	0.00170	30.25	15.62	0.2379	0.00758
6.0	0.00121	42.54	21.72	0.3348	0.00758
7.0	0.00086	59.89	30.11	0.4554	0.00758
8.0	0.00061	84.38	42.07	0.6417	0.00758
9.0	0.00043	118.81	59.85	0.9207	0.00758
10.0	0.00031	167.26	83.07	1.2834	0.00758
11.0	0.00022	235.56	119.18	1.8414	0.00758
12.0	0.00015	331.93	161.25	2.4913	0.00758
13.0	0.00011	467.92	227.45	3.4314	0.00758
14.0	0.00008	659.90	341.18	5.1471	0.00758
15.0	0.00005	931.03	454.90	6.8627	0.00758
15.9	0.00004	1269.53	682.35	10.2941	0.00758

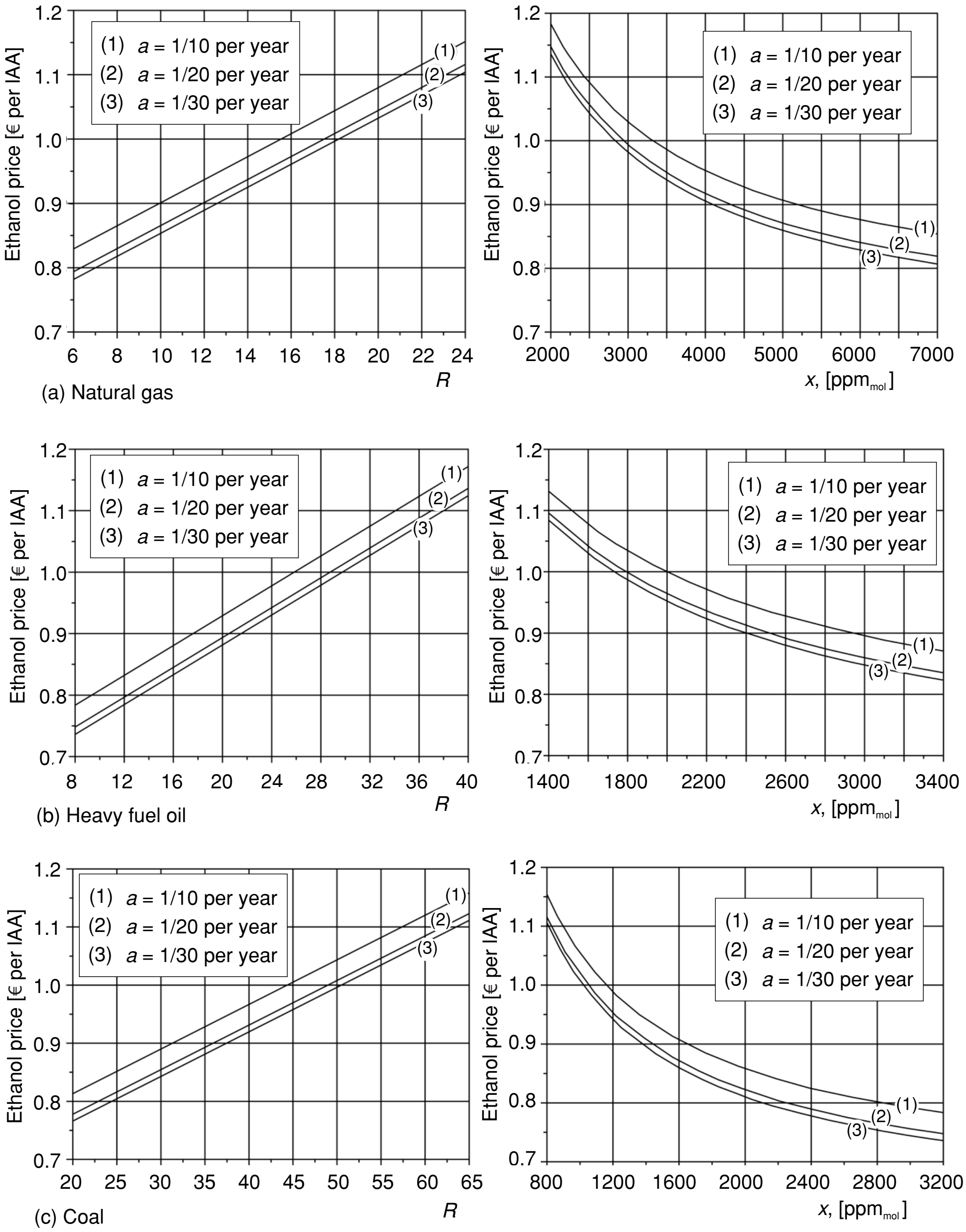

Figure 2. Production cost of ethanol during exhaustion

In order to establish a proper mathematical model for material and energy balance we have conducted the field measurements on industrial rectification plant in Kostojevici. These measurements included monitoring of all important working parameters such as distillate volume flow rate, consumption of steam, reflux ratio, consumption of cooling water, as well as the monitoring of composition of the distillate and liquid in the reboiler. It was concluded that the normalized tray efficiency model presented in [12] provides the satisfactory accuracy, higher than some other models (AICHE and others).

Until the exhaustion begins average consumption of steam is $s_{\mathrm{HS,av}}$ = 2.4157 kg per LAA and average consumption of cooling tower water is $s_{\mathrm{CW,av}}$ = 0.09808 m^3 per LAA.

Current specific consumption of water from well during exhaustion is practically regardless of the time and it is equal to $s_{\mathrm{well}}(\tau)$ = 0.00785 m^3 per LAA.

Average consumption of water from well until the moment of the exhaustion beginning is $s_{\mathrm{well,av}}$ = 0.00681 m^3 per LAA.

According to *profit production criterion* distillate production is interrupted at the moment when the selling price of ethanol equalize with the total production cost, which can be represented by the equation:

$$C_{\mathrm{EP}}=\frac{aC_{\mathrm{inv}}}{V_{D,\mathrm{AA,year}}}+\left[s_{\mathrm{HS}}\left(\tau\right)+s_{\mathrm{HS,av}}\right]C_{\mathrm{HS}}+\left[s_{\mathrm{CW}}\left(\tau\right)+s_{\mathrm{CW,av}}\right]C_{\mathrm{CW}}+\left[s_{\mathrm{well}}\left(\tau\right)+s_{\mathrm{well,av}}\right]C_{\mathrm{well}}+\left(C_{\mathrm{tot}}^{\mathrm{AA}}\right)_{\mathrm{I}} \tag{6}$$

The distillery was originally designed to use steam produced in fuel oil boiler. In that case, using eq. (5), it was calculated that, depending on the amortization rate, the exhaustion is economically justified up to the to moment τ = 3.78-4.30 hour, which corresponds a reflux ratio R = 20.03-23.86 and the ethanol content in the residue 2580-2160 ppm$_{\mathrm{mol}}$. As shown in tab. 5, slightly different values were obtained when other fuels (natural gas or coal) are used. Cheaper fuel (coal) extends the duration of exhaustion allowing the greater exhaustion of liquid in the reboiler (reducing the ethanol content until 1470-1250 ppm$_{\mathrm{mol}}$) and increasing reflux ratio to R = 35.06-41.11. That's why the boiler for solid fuel (coal) is subsequently embedded in the plant, making it possible to decrease production cost by 12.9% [13].

Table 5. Profitability of exhaustion *vs*. fuel type and amortization rate - limit values

	Natural gas			Heavy fuel oil			Coal		
Amortization rate, year^{-1}	1/10	1/20	1/30	1/10	1/20	1/30	1/10	1/20	1/30
Reflux ratio	11.63	13.61	14.23	20.03	22.93	23.86	35.06	39.51	41.11
x_{W}, [kmol/kmol]	4440	3800	3630	2580	2250	2160	1470	1300	1250
τ, [hour]	2.17	2.63	2.77	3.78	4.18	4.40	5.43	5.78	5.90

Using the current prices in Serbia, natural gas is the most expensive fuel, which leads to a reduction in the duration of exhaustion and to increase the content of ethanol in the reboiler until 4440-3630 ppm$_{\mathrm{mol}}$. In this case, the process should be stopped at a reflux ratio R = 11.63-14.23.

Figure 2 shows the change of the production cost of ethanol during the exhaustion process depending on the ethanol content in the residue in the reboiler, (*i. e*. from reflux ratio), fuel type and the amortization rate. Knowing the selling price of ethanol it is possible to use these charts to determine the moment when the process of the exhaustion should be stopped, since it becomes economically unjustified. It should be noted that this ethanol production cost refers only to the ethanol produced after the tail fractions are removed.

Comment of the results and their comparison with the values measured at industrial plant

The composition of the main product of distillery (hart fraction) is presented in tab. 6 (ethanol fraction was determined by densitometry, acids were determined by volumetric titration, while other components were detected using a gas chromatograph Unicam ProGC with Flame Ionization Detector). Table 6 also contains the compound fractions in distilate according to the Serbian standard SRPS E.M3.020 and appropriate European Regulation (EC) No. 110/2008. Analyzing these compositions, it can be concluded that the distillate meets local standard, but according to European standard the content of fusel oils is higher than the permitted value. The fusel oils can be separated from bottom trays of the rectification column. Separation of fusel oils is provided as an option on the rectification column, but since the conditions prescribed by local standard are satisfied the manufacturer did not run this separation sequence.

Table 6. Comparison of the distillate compositions with the demand composition from local and European standard

i	Component	h_{Di}	SRPS E. M3.020	Regulation (EC) No 110/2008	Unit
1	Ethanol	0.859695	0.857670	0.857670	kmol/kmol
2	Aldehydes (acetaldehyde)	0	35.4	5.6	ppm_{mol}
3	Methanol	16	1930.8	468.1	ppm_{mol}
4	Esters (ethyl acetate)	5	-	7.4	ppm_{mol}
5	Fusel oil	23	23	3.4	ppm_{mol}
	5.1 isopropanol 5.2 isobutanol 5.3 isoamyl alcohol	9 9 5	-	-	ppm_{mol}
6	Acids (acetic acid)	13	50	12	ppm_{mol}
7	Water	0.140248	0.140290	0.142970	kmol/kmol

We have also measured the ethanol content in liquid remained in the reboiler at the end of the exhaustion and the measured value was 1804 ppm_{mol} (steam boiler used coal as a fuel). Obviously this value is higher then calculated minimal value (1470-1250 ppm_{mol}) and corresponds to a reflux ratio R = 28 which is the upper limit recommended in [3]. Having in mind the calculated values we have advised the manufacturer to extend the exhaustion period.

Conclusions

In this paper we have presented the analysis of work of batch rectification plant for ethanol production. After hearth fraction separation it is no longer possible to obtain the distilate with desired properties, but the content of ethanol in the reboiler is still significant. That is why the process should be continued in order to maximize utilization of ethanol. The aim of the analysis was to determine the extent to which it is economically viable to exhaust the residual liquid in the reboiler, and the *profit production* criterion is used for calculations. According to this criterion production of distillate has to be interrupted at the moment when the selling price of ethanol equalizes with the total production cost, which is represented by the eq. (6). For this purpose fig. 2 can be used, and it shows the change of the production cost of ethanol during the exhaustion process depending on the ethanol content in the residue in the reboiler, (*i. e.* from reflux ratio), fuel type and the amortization rate. When the market price of rectified ethanol is known, it is possible to use these charts to determine the moment when the process of the exhaustion should be stopped, since it becomes economically unjustified. It should be noted that this ethanol production cost refers only to the ethanol getting from residue after tail fractions removing. For specific rectification plant built in village Kostojevici in Serbia, and using the current utility prices in Serbia, the range of reflux ratio at the end of the process is R = 12-1. Appropriate ethanol content in the residue is in range 4440-1250 ppm_{mol}, and these values strongly depend on the fuel type and amortization rate (see tab. 5). It has to be noted that ethanol fraction in residue is much higher then for continuous distillation plant (250-300 ppm_{mol}), as shown in [2].

Nomenclature

a – amortization rate, [$year^{-1}$]
C – cost, [€], [€ per m^3], [€ per LAA], [€ per kg]
D – mass flow rate of distillate, [kgh^{-1}]
F – factor, [–]
f_i – direct-cost factors (equipment erection, piping, electrical power, instruments, process buildings, storages, utilities, site preparation, *etc.*), (i = 1-9), [–]
$\dot{m}$ – mass flow, [kg per hour]
N_R – number (quantity), [–]
R – reflux ratio, [–]
s – specific consumption, [kg per LAA], [m^3 per LAA]
t – temperature, [°C]
$\dot{V}$ – volumetric flow rate, [m^3 per hour], [L per hour], [LAA per hour], [LAA per day]
x – mole fraction of component i, [$kmol_i$/kmol]

Greek symbols

$\varepsilon_{D,TEF}$ – volume fraction of ethanol in the distillate during exhaustion, [LAA per litre]
τ – time (duration of the exhaustion), [hour]

Subscripts

av – average
B – bare module
BE – major process units (basic equipment)
Col – column
CW – water from cooling tower
D – distillate
EP – economic profitability
HE – heat exchanger
HS – heating steam
I – continious distillation
inv – investment
M – material
nom – nominal
op – operating
P – pressure
SC – shell of distillation column
T – tray
t – type
tot – total
W – residue
wb – wet bulb
well – water from well

Superscripts

AA – absolute alcohol (100% ethanol)

Acronyms

HF – head fraction
TEF – tail exausting fraction
TF – tail fraction

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