

Potential of Tannery Fleshings in Biodiesel Production and Mathematical Modeling of the Fleshing Pre-Treatment

Karel Kolomaznik, Jiri Pecha, Michaela Barinova, Lubomir Sanek, Tomas Furst, and Dagmar Janacova

Abstract— Waste fat produced by tanneries during the processes in which raw hide is transformed into leather represents a very important raw material for biodiesel production. However, the acid number of this feedstock is usually high above the limit 1 mg KOH/g, which makes it unsuitable for direct processing into biodiesel via alkali-catalyzed transesterification. An effective and economically viable pre-treatment method is needed to overcome this disadvantage and at the same time to maintain the advantage of low price of the feedstock. In this paper, we present a pre-treatment method consisting of the refining melting and deacidification steps. The pre-treatment processes are part of a complex technology for biodiesel production from low quality fats and oils. Mathematical models of the said processes have been elaborated for the optimization of the pre-treatment procedure, as well as a software application developed for simulation of the deacidification step. Suitability and economic parameters of the pre-treatment technology for feedstocks with various free fatty acid content is discussed. Finally, the waste tannery fat potential in biodiesel production is briefly evaluated from the economic and raw material base point of view.

Keywords— Biodiesel, animal fleshings, pre-treatment, mathematical modeling.

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I. INTRODUCTION

SOYBEAN oil is the predominant feedstock for biodiesel production in the USA, while rapeseed oil prevails in Europe. The high price of biodiesel (over double the price of diesel) mostly results from the price of the feedstock, which makes up to 75% of the overall production costs [1]. However, biodiesel can be made from other feedstocks, such as beef tallow, pork lard, yellow grease and waste tannery fats [2, 3]. Especially the latter kind of waste fats is produced in large quantities and should be taken into account as a promising feedstock for biodiesel production [4]. They usually represent an unpleasant waste and are consequently incinerated. The incineration is charged, which makes them a very cheap feedstock for fuel industry.

However, there are certain limitations which should be taken into account to make the biodiesel production economically viable. First, water, nitrogen and ash must be removed prior to further processing. This can be done via various rendering methods. The obtained fat though contains considerable amount of free fatty acids (FFA) which makes it unsuitable for the subsequent alkali catalyzed transesterification. The acid value of fleshings is usually high above 1 mg KOH/g, which is according to [5] the limit FFA content to obtain commercial grade biodiesel that meets the American Standard ASTM D 6751. This makes the fleshings practically impossible for direct conversion into biodiesel.

Efforts have been made to overcome this problem and develop a method of direct utilization of acidic fats in biodiesel production. Supercritical methanol processes are being developed in order to convert both FFA and triglycerides to biodiesel [6]. This method though requires special equipment for it employs high pressure and temperatures. Enzymatic methods of waste fat and oil utilization in biodiesel production have been described [7]; however, they are rather in the stage of laboratory testing. As a result, most industrial processes use alkali catalyzed transesterification with a preceding FFA removal step.

There are several methods of deacidification used in industrial scale. The chemical one most frequently employs sodium hydroxide to neutralize the FFA, the physical method strips the FFA with steam under vacuum. The FFA content can

also be reduced by an extraction with a suitable solvent (e.g. methanol) [8]. Probably the most investigated way is an acid-catalyzed esterification of FFA [9]. The method uses a strong inorganic acid (such as sulfuric acid) for esterification of the FFA to methyl esters. The esterification reduces the FFA content to a level at which an alkali catalyst may be employed for subsequent transesterification of acylglycerols [10]. In [11] is used a scale-up of the two-step esterification technology in which sulfuric acid was employed as an esterification catalyst and the refined fat was transesterified with an inorganic alkali.

The main drawback of the described techniques is the necessity of acid neutralization prior to alkali transesterification; this leads to a formation of large amounts of salts in the reaction system [12]. These salts must be removed from the final biodiesel (and also glycerin) which may be the limiting factor in the product purification [13]. Moreover, the method is also complicated with a necessity of a subsequent removal of water formed during the reaction [14]. Much effort has been made to develop suitable solid esterification catalysts (e.g. [12]) which are easily separable from the reaction mixture after successful esterification. Despite the fact that some of these solid catalysts are available commercially, the solid catalyst efficiency and its possible fouling are still an issue, namely in case of esterification under mild reaction conditions, i.e. at atmospheric pressure and temperatures around 60 °C.

Our contribution addresses the key tasks of acidic tannery fleshing pre-treatment, namely an efficient rendering and deacidification. Mathematical models of the said processes are presented, as well as a software application developed to simulate the deacidification step. Last but not least, the paper gives an evaluation of the waste tannery fat economical potential in biodiesel production.

II. THEORY

A. Mathematical model of the zonal refining melting

Mathematical modeling as a main tool of chemical engineering proved useful in many areas of chemical industry. For the purposes of our study, we proceeded particularly from the mathematical description of chemical-physical processes taking place in the fleshing material. Transport phenomena connected to collagen material desalting are described e.g. in [15, 16]. The desalting process is not part of our contribution; however, it is applied to fleshings coming from cured hides prior to the processes described below. To make the waste tannery fat usable for biodiesel production, it is necessary to remove water, reduce the ash content and, if possible, to reduce free acid content (acid number). We applied the refining melting process. First, the fat is heated to the melting point. Due to the low value of melting point (about 50°C) of the waste fat, we can use the waste heat from a dryer. The general mathematical-physical model of the process is represented by the following equations:

$$\frac{\partial t}{\partial \tau}(\tau, \hat{x}) = a \Delta_{(\hat{x})} t(\tau, \hat{x}) \quad (1)$$

$$t(o, \hat{x}) = t_p \quad (2)$$

$$t(\tau, \hat{x}) = t_o(\tau) \text{ for } \hat{x} \leftarrow \partial\Omega \quad (3)$$

$$\dot{Q} = B \frac{d \cdot t_o(\tau)}{d\tau} + C \frac{\partial t}{\partial \hat{n}}(\tau, \hat{x}) \quad \hat{x} \leftarrow \partial\Omega \quad (4)$$

$$B = V_0 \rho_0 c_{p0} \text{ and } C = \lambda S \quad (5)$$

where (1) describes non-stationary temperature field, $\Delta_{(\hat{x})}$ is the Laplace operator transformed into the respective coordinates, (2) is the initial condition, (3) represents the border of the sample surface set, (4) describes the heat balance on the heated sample surface and \hat{n} stands for the outer unit normal vector. As an example of solution for the above mentioned general model, dimensionless non-stationary temperature field for a cylinder shape is presented:

$$T = \frac{t}{t_p} = 1 + \frac{1}{16} + \frac{A}{4} + \frac{R^2}{4A+2} + 2P \sum_{n=1}^{\infty} \frac{J_0(R q_n) \exp(-Fo q_n^2)}{q_n^3 A J_1(q_n) - q_n^2 J_0(q_n) + 2q_n J_1(q_n)} \quad (6)$$

where q_n are roots of the following equation:

$$A J_0(q) + \frac{J_1(q)}{q} = 0 \quad (7)$$

and

$$R = \frac{r}{b}; P = \frac{\dot{Q} b^2}{2V t_p \lambda}; A = \frac{a V_0 c_{p0} \rho_0}{2V \rho} \quad (8)$$

If the cylinder diameter exceeds by ten times or more the thickness of the melting layer (which condition is usually fulfilled, especially in the beginning of the process), the model can be approximated to an infinite plate.

The proposed model proceeds from several assumptions: it can be well-approximated to a one-dimensional heat transfer model, with laminar flow. At the same time, the melting process runs evenly along the plate; the only changing parameters are the thickness of the melted-down layer and its surface temperature along the horizontal dimension. The model geometry is depicted in Fig. 1, the dependence of the fat layer thickness on the horizontal position in Fig. 2.

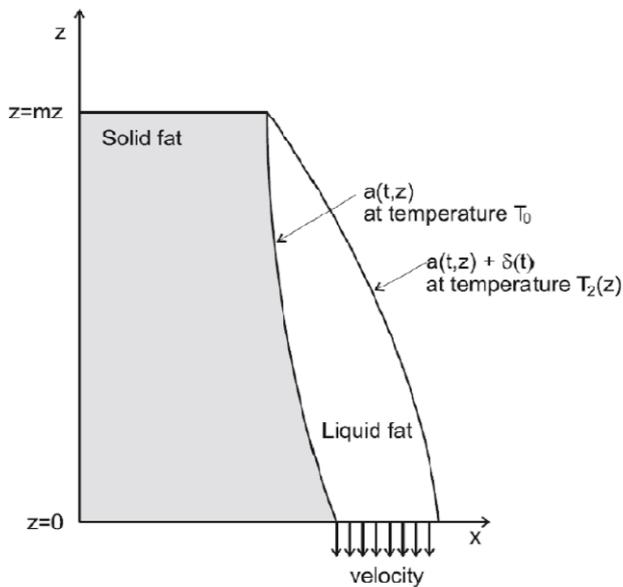


Fig. 1 Geometry of the mathematical model of zonal refining melting.

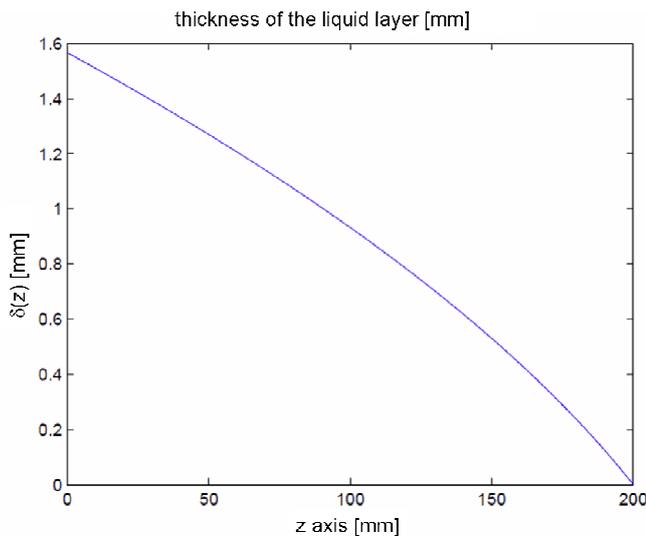


Fig. 2 Dependence of the liquid fat layer thickness on the horizontal position.

The next procedure depends on the FFA content. If too high, it is necessary to implement the esterification. We carried out esterification of the FFA in alkali conditions of tetramethylammonium hydroxide (TMAH). The main advantages of using of organic bases lie in the fact that no ballast inorganic salts are produced, high quality glycerin is obtained and the following fat reesterification by methanol can be performed immediately. Another asset is that the said volatile organic bases can be partially regenerated during removal of the excess methyl alcohol by distillation.

B. Mathematical model of deacidification (extraction) step

Solvent and liquid fat are mixed in the extraction reactor. The heterogeneous system is stirred continuously so that the mass transfer between particular phases can take place. After the stirring is stopped, the extractor acts as a separator containing F kg of the mixture, which is subsequently separated into M kg of the solvent phase (extract) and O kg of the fat phase (raffinate). Partition coefficients K_i were determined experimentally. The partition coefficient is defined as a ratio of an equilibrium mass fraction of the i -th compound in the solvent (a_{iM}) and fat (a_{iO}) layer and is the basic and most important input for the computing algorithm used for the optimization of the extraction process.

$$K_i = \frac{a_{iM}}{a_{iO}} \quad (9)$$

The total mass balance of the separator is defined by the following equation:

$$F = M + O \quad (10)$$

Then, the mass balance of the i -th component is:

$$f_i = m_i + o_i \quad (11)$$

Consequently:

$$F = \sum_{i=1}^n f_i; M = \sum_{i=1}^n m_i; O = \sum_{i=1}^n o_i \quad (12a,b,c)$$

$$a_{iM} = \frac{m_i}{M}; a_{iO} = \frac{o_i}{O}; a_{iF} = \frac{f_i}{F} \quad (13a,b,c)$$

Substitution of (13a,b) into (9) gives:

$$K_i = \frac{m_i \cdot O}{M \cdot o_i} \Rightarrow o_i = \frac{m_i \cdot O}{K_i \cdot M} \quad (14a,b)$$

Finally, by using (14b) together with (11) we get:

$$m_i = \frac{f_i}{1 + \frac{O}{M} \cdot \frac{1}{K_i}} \quad (15)$$

C. Software for the optimization of the deacidification (extraction) step

Computer simulation of the modeled processes allows processing of complex data sets and provides quick determination of the process behavior with various input data. We based the software development on our previous studies

relating to simulation of industrial processes, published e.g. in [17].

The above described mathematical model is solved with the use of an iterative method, particularly an approximate balance method, which is a variant of a fixed-point theorem in the space of concentrations. This method represents the core of the software developed for the optimization of the extraction process. The calculation of the approximate balance itself is performed using the bisection method. The initial mixture of feed and solvent (extraction agent) is separated into two phases: extract and raffinate. The calculation is based on experimentally measured partition constants which are defined by the user. The other inputs are composition of the feed and the amounts of both the feed and solvent. The software is able to calculate the composition of the final raffinate and extract, the ratio of the raffinate to solvent. In case of fat deacidification, the software also calculates the deacidification efficiency, loss of fat (more precisely triacylglycerols) in the extract, and loss of the solvent in the raffinate (all in one step). A better insight into the developed software is given in Fig. 3 and Table I. The figure represents the dialog window for the input data; the table gives a set of the resulting calculations.

D. Economy of the waste fat pre-treatment employing an esterification agent

The purpose of the waste fat and oil pre-treatment is to achieve the feedstock quality comparable to that of pure vegetable oils. The economic viability of the biodiesel production process from waste fats and oils is determined by the costs of the pre-treatment processes. The total costs of the pretreatment (including the price of the waste fats) must be lower than the price of pure vegetable oils. This approach enables us to apply relatively simple mathematical models based on economic balance because it is not necessary to deal with transesterification process itself which is not different from the common technology. The costs of water removal and refining melting are low (waste heat of tannery driers may be employed for this purpose) in comparison to the esterification of FFA. Thus, we presume that only costs of esterification together with price of the feedstock are substantial for the economic optimization of the pre-treatment technology.

The specific price of waste fats and oils (n) may be represented as a function of their FFA content which is expressed as an acid value (x). It is possible to approximate the real data with the exponential cost function (16).

$$n = A \cdot \exp(B \cdot x) + C \quad (16)$$

The price of the esterification agent is dependent on the actual feedstock acidity value, thus the overall operating costs (y) can be written as (17).

$$y = K_{OB} \cdot x + n \quad (17)$$

According to our presumptions, the profit (p) is then equal to

the difference between the price of pure vegetable oil and the overall operating costs of waste fat pre-treatment (18).

$$p = k = y \quad (18)$$

The extrema ($X_{min,max}$) of the above stated functions – the minimum of the overall operating costs and maximum of the profit – can be calculated from (19).

$$X_{min,max} = \frac{-1}{B} \ln\left(\frac{K_{OB}}{A \cdot B}\right) \quad (19)$$

There is a certain limiting factor of biodiesel production from waste animal fats – the cloud point of such biodiesel is higher than the cloud point of biodiesel made of vegetable feedstock. This is caused by the fact that the melting point of saturated fatty acid methyl esters is higher in comparison with the methyl esters of unsaturated fatty acids. The said disadvantage may be reduced by blending of waste animal fats with waste vegetable oils, such as used cooking oils. The key factor, again, is the economic viability of this method. It is therefore necessary to ensure that the overall operating costs of the pre-treatment procedure do not exceed the acceptable level. Since the tannery fat is at least on the Czech market free of charge (at current state), we may assume that the overall operating costs (y) are determined by the costs of esterification of FFA present in both waste tannery fat and waste vegetable oil, and the price of waste vegetable oil n_O – see (20).

$$y = K_{OB} [x_{TF} \cdot a_{TF} + x_O \cdot (1 - a_{TF})] + (1 - a_{TF}) \cdot n_O \quad (20)$$

III. EXPERIMENTAL

A. Zonal refining melting

10 kg of waste fat were weighed out, placed into a linen sack and then put into a reheating furnace. The initial temperature was approximately 20°C and the liquefaction occurred at about 40°C (the melting point). At the same time, water steam was exhausted. The melting lasted 2 hours and 7 kg of the raffinate were obtained. The following Table II shows the composition of the raffinate in comparison with the composition of the feedstock before the refining process.

Names of the substances	Mass fractions in the crude fat	Partition coefficients methanol : oil	Total mass of the input [kg]
Fat	0.77	0.08	15.00
MeOH	0.00	15.00	10.00
Water	0.02	7.80	
Base	0.00	0.00	
Soap	0.20	8.80	
Acids	0.01	1.90	
blank	0.00	0.00	
blank	0.00	0.00	
Total	1		

Parameters of chemical deacidification

Weight of the base	0.03094	<input checked="" type="checkbox"/> Ekvimolarne
Molar mass of the base [g/mol]	56.11	
Molar mass of the acid [g/mol]	272	

Fig. 3 The dialog window for the input data.

Table I Set of the calculation results obtained from the software.

Component	Input:oil [1]	Part. coeff. [1]	Output:oil [1]	Output:oil [kg]	Output:MeOH [1]	Output:MeOH [kg]
Fat	0.77	0.08	0.92462	10.4594	0.07397	1.0906
MeOH	0	15	0.043017	0.48662	0.64526	9.5134
Water	0.02	7.8	0.0040268	0.045552	0.031409	0.46308
Soap	0	8.8	0.028336	0.32055	0.24936	3.6764
Acids	0.21	1.9	0	0	0	0
Total	1		1	11.3122	1	14.7434

Ratio O/M = 0.76727
 Deacidification efficiency = 0.9198
 Fat loss = 0.094421
 Methanol loss = 0.048662
 Input mass of crude fat = 15 kg
 Input mass of methanol = 10 kg
 Input mass of base = 1.0556 kg
 Molar mass of base = 91.15 g/mol
 Molar mass of acid = 272 g/mol

Table II Comparative composition of the fat refining products.

	refined	non-refined
Volatile matter at 103°C (%)	undetected	27.26
Nitrogen (TKN)	undetected	undetected
Melting point (°C)	19.9	34.2
Ash (%)	0.16	1.05
Saponification number (mg KOH/g sample)	181.4	128.9
Acidity number (mg KOH/g sample)	10.6	13.3

The table shows considerable decrease in ash content and a slight decrease in acidity number in the refined product. All the moisture was practically removed during the refining melting.

B. Biodiesel synthesis from refined fat

The refined fat was extracted with methanol (50 % w/w) which contained equimolar amount of tetramethylammonium hydroxide (TMAH) (with respect to the FFA content). The mixture was left to settle and the extract and deacidified fat were collected. The deacidified fat was then loaded into glass reactor with methanol and adequate amount of TMAH methanolic solution. The reaction mixture was then refluxed and stirred for two hours. Next, it was transferred into a separatory funnel where the clear separation of hot glycerin and methyl ester (FAME) layers was observed. The layers were cooled at laboratory temperature and separated. The FAME layer was further distilled in a vacuum rotary evaporator and a sample of taken and subjected to analysis.

C. Determination of fatty acid methyl esters

About 250 mg of the FAME sample is weighed into a vial and 5 ml of the storage solution of the inner standard C17 is added (methyl heptadecanoate) for FAME quantification. Chromatographic conditions: Column: w/Integra-Guard (30 m × 0.32 mm × 0.25 μm). Heat program of the furnace: 210°C (kept for 5 min), then warming up to 230°C @ 20°C/min, the temperature was again kept for 5 min. Injector: PTV, manual injection 1 μl in the split mode (split ratio 1:100), and temperature of the injector 250°C. Detector: FID, temperature 250°C, limit of the detector detection (Range) = 10. Hydrogen was used as a carrier gas, with a constant flow rate of 3 ml/min. Duration of the analysis: 11 min. The resulting chromatogram can be seen in Fig. 4.

IV. DISCUSSION

A. Zonal refining melting

The semi-pilot experiment has shown very good results of the zonal refining melting and proved to be more effective than the conventional melting process. The laboratory and semi-pilot tests definitely confirmed the necessity of the pre-treatment in the feedstock containing high portion of water and non-fatty components (e.g. hair and skin splits in tannery fats). The transesterification conversion of a non-refined

feedstock is very low, making the process economically unsustainable. However, the crucial factors determining the processing technology are the technological costs while maintaining the minimal output requirements. From this point of view, the zonal refining melting process is not absolutely necessary for the biodiesel production process. The acid number of long-stored fats is very high compared to the fresh waste fats. This implies that it is economically profitable to process the waste fats as soon as possible, because it considerably reduces the pre-treatment costs as well as the technological demands of the whole biodiesel production process. In some waste feedstock, the pre-treatment technology can be avoided at all, because the feedstock contains very low amount of FFA and has practically zero water and non-fat component content. This is for example the case of some used cooking oils.

B. Deacidification

Common way of free fatty acids removal lies in their acid catalyzed esterification. Since this reaction is reversible, it is necessary to use an excess of alcohol to achieve high conversion of the esterification step. In addition to that, if acidic waste fat is used (such as brown grease) instead of pure FFA, significant losses of methyl esters occur during the resulting biodiesel washing phase. This was reported in [5] during the experiments done in a pilot plant. The deacidification method presented in this paper does not suffer from the above mentioned complications. The physical extraction of FFA (with pure methanol used as an extraction agent in our case) is suitable rather for feedstock with low acidity; nevertheless, also in such case a multistage extraction process may be necessary. On the other hand, extraction with chemical reaction (using methanolic solution of TMAH) is able to deacidify the feedstock in one stage because the organic soaps formed during the neutralization reaction are mostly transported to the methanol phase. The low amount of FFA and soaps present in the resulting deacidified fat facilitate easy separation of glycerin and methyl ester layers after the subsequent transesterification.

C. Economic aspects of the waste fat pre-treatment with esterification agent

To determine and analyze the appropriate cost functions we proceeded from our long-term experience with optimization of technological processes, published for example in [18]. Since the dependency of the feedstock price on its acid value is non-linear, the operating cost (17) and profit (18) functions have extrema. Fig. 3 reveals their position on the Czech market.

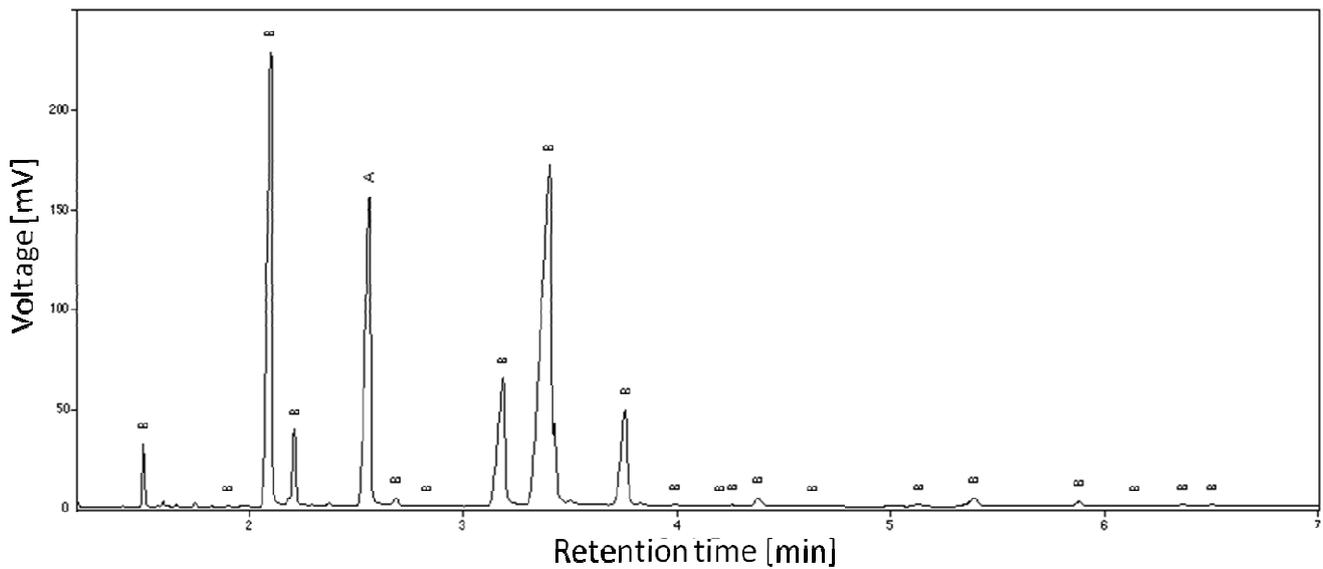


Fig. 4 Chromatogram of the FAME sample. The "A" peaks stand for the inner standard C17, the "B" peaks represent the fatty acid methyl esters.

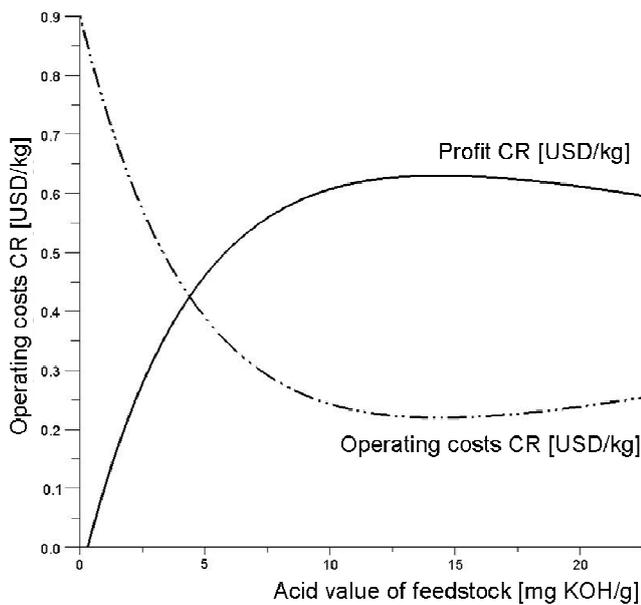


Fig. 5 Estimated profit and operating costs as functions of feedstock acid value (Czech market).

The figure shows that the maximal profit over 0.6 USD/kg is obtained when the feedstock has acid value around 15 mg KOH/g (the profit estimation is valid for the catalyst price 0.008 USD/acid value). Aside from the price of the waste fats and oils, the second important constituent of the overall operating costs is the price of the esterification agent. Since the esterification agent is consumed during FFA pre-treatment, the maximal profit moves forward to the feedstock with lower acid value when the price of the organic base is increased. An

optimal acidity value of the feedstock (which ensures maximal profit) plotted as a function of the alkylation agent price in Fig. 6 reveals clearly this relation. However, the acid values of the feedstock often fluctuate in the reality. In such case an optimal composition can be found with the help of (20).

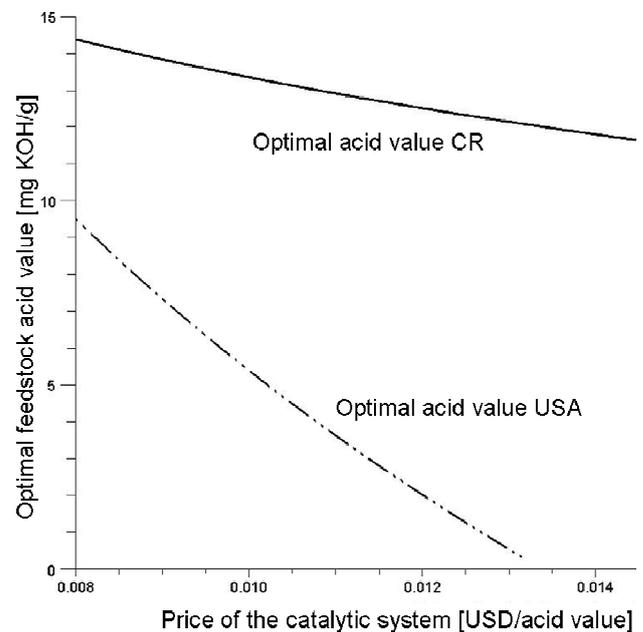


Fig. 6 Evaluated optimal feedstock acid value and its dependency on the price of the catalytic system.

D. Economic potential and raw material base of tannery fats for biodiesel production

The net calorific value of pure fat is approximately 39

MJ/kg. An average price of heat is about 20 USD/GJ (Czech Republic). The following Table III shows the earnings of various ways of tannery waste fat utilization:

Table III Economic income from various ways of waste tannery fat utilization.

Way of processing	Unit price of power	Economic income /kg fat
Heat	20 USD/GJ	0.78 USD/kg
Electric power	0.25 USD/kWh	0.68 USD/kg
Methane	0.5 USD/m ³	0.5 USD/kg
Biodiesel	2.4 USD/l	2.4 USD/kg
Glycerin	0.5 USD/kg	0.05 USD/kg

Although the real market prices of waste fat processing products may differ, their relative comparison clearly shows that from the economic point of view biodiesel production is the most profitable way. The raw material base is wide in global scale and according to current trends it is not expected to be reduced. Animal fleshings represent 50-60% of the total waste generated by the leather industry [4]. An estimated fleshing generation worldwide is approximately 3.8 million tons according to [19]. It should be emphasized that our calculations proceed from available data e.g. from tanneries. In other words, the real material base can be substantially wider.

V. CONCLUSION

Our current research has confirmed the feasibility of using waste tannery fats in biodiesel production. There are two key steps involved in the pre-treatment process – an effective refining melting and free fatty acids removal (deacidification). Via the refining melting it is possible to considerably decrease the content of water, ash, the acid number, or, possibly, the nitrogen content. The deacidification is performed via a physical process of extraction with methanol as the extraction agent. However, for feedstock with high acidity it may be necessary to perform extraction with a chemical reaction. For the esterification of free fatty acids it is beneficial to use alkali conditions achieved with the use of organic bases, e.g. tetramethylammonium hydroxide. The excess methylalcohol and the said organic bases can be re-distilled and reused in the reesterification reaction. Mathematical models of the zonal refining melting, deacidification and the economical aspects of the pre-treatment technology are presented. The effectiveness of the pre-treatment process as well as the consequent transesterification of the refined fat have been experimentally proved on a semi-pilot scale. Finally, waste tannery fat potential in biodiesel production has been evaluated from the economic and raw material base point of view.

VI. LIST OF SYMBOLS

t	Temperature	[K]
τ	Time	[s]
x	Coordinate	[1]

t_0	Surrounding temperature	[K]
V_0	Surrounding volume	[m ³]
ρ_0	Surrounding density	[kg.m ⁻³]
$c_p\theta$	Specific heat of the surrounding	[J.kg ⁻¹ K ⁻¹]
$J_0(q)$	Bessel function of zero order	[1]
$J_1(q)$	Bessel function of the first order	[1]
λ	Thermal conductivity coefficient	[W.m ⁻¹ K ⁻¹]
S	Surface of the heated sample	[m ²]
\dot{Q}	Thermal power of the heater	[W]
b	Sample thickness	[m]
V	Sample volume	[m ³]
t_p	Sample initial temperature	[K]
a	Thermal conductivity coefficient of the sample	[m ² .s ⁻¹]
a_{TF}	Mass fraction of the tannery waste fat	[1]
A	Parameter of feedstock cost function	[USD/kg]
B	Parameter of feedstock cost function	[g/mg KOH]
C	Parameter of feedstock cost function	[USD/kg]
k	Price of pure vegetable oil	[USD/kg]
K_{OB}	Price of organic base catalyst/esterification agent	[USD/acid value]
n, n_0	Specific price of the feedstock and waste oil, respectively	[USD/kg]
p	Profit	[USD/kg]
x, x_{TF}	Acid value of the feedstock, tannery fat and oil, respectively	[mg KOH /g]
x_0		
$X_{min,max}$	Extrema of the function	
y	Overall operating costs	[USD/kg]

Other symbols are explained directly in the text.

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