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Evaluation of new biodegradable fluid on the basis of accelerated durability test, FTIR and ICP spectroscopy

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Abstract

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The paper deals with the properties of newly developed synthetic biodegradable fluid in terms of its operational and physico-chemical properties. The evaluated fluid is a new biodegradable fluid MOL Farm UTTO Synt, which belongs to the group of universal gear-hydraulic fluids. At the beginning, the fluid was subjected to accelerated durability test under laboratory conditions, with monitoring its impact on technical conditions of the used hydraulic pump UD 25. After that, the FTIR spectroscopy analysis was performed, with monitoring thermal oxidation, oxidation by acid products, water content and additives depletion. Depletion of additives was also monitored by the ICP spectroscopy. Based on these results, it can be stated that the newly developed fluid has good operational and physico-chemical properties after the durability test and is suitable for further testing under operational conditions without a risk of damaging agricultural machines.

Keywords: new gear-hydraulic fluid; hydrostatic pump; aditives; physico-chemical properties

More and more difficult requirements in terms of environmental protection are imposed on machines working in the agricultural sector. All agricultural machines need working medium for operation of their working and handling equipment. In majority, this working medium is hydraulic fluid.

Similarly, with tightening legislation, increasingly greater requirements are imposed on hydraulic fluids in terms of their operational properties and impact on the environment. The trend in the application of hydraulic fluids indicates replacement of traditionally produced fluids, most of which have low biodegradation and high ecotoxicity, with plant-based fluids (Tkáč et al. 2010). In agriculture, machines are in direct contact with the environment where foodstuffs originate. Whether agricultural land is used for growing food or forage, ultimately all contaminants have an effect on the quality of final product. Therefore, the solution of problems associated with agricultural ecology is up to date and highly helpful for society. Almost 50% of all the oils sold in the world finish at present times as residues during the operation in nature (Jakob et al. 2006). The right choice of working medium has also a high impact on a failure-free operation. Momentary, hydraulic systems of mobile machinery use mainly mineral oils having good properties proven by many years of use (Majdan et al. 2012). Most current lubricants contain petroleum base stocks, which are toxic to environment and difficult to dispose of after use (Kučera, Ruesek 2011). However, a slow but steady move towards the use of environmentally friendly or more readily biodegradable lubricant fluids has taken place during the last decade. Biodegradability has become one of the most important design parameters both in the selection of base fluids and in the overall formulation of the finished lubricant (Mendoza et al. 2011). Many equipment

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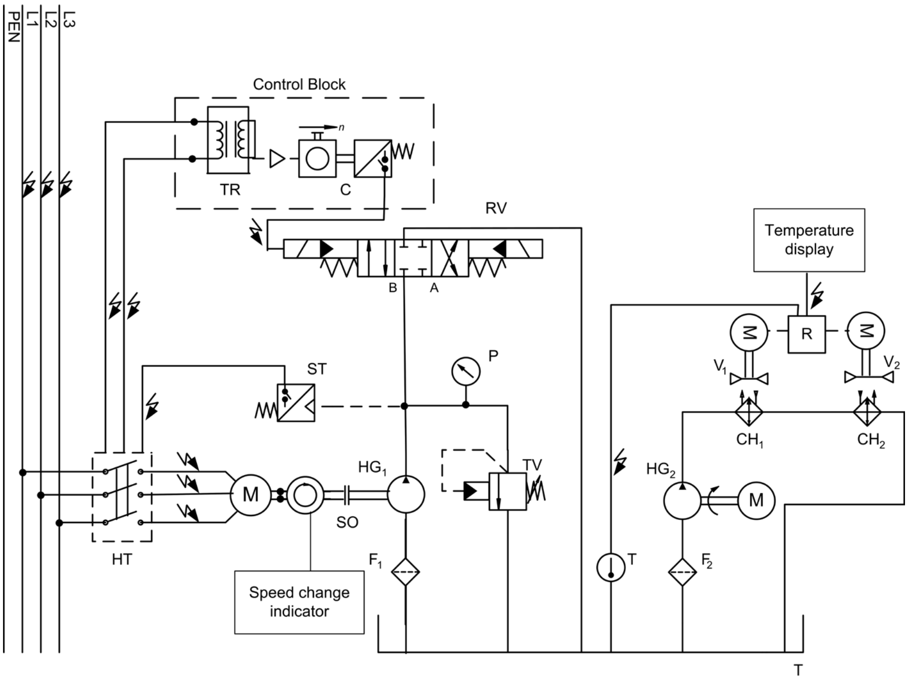

Fig. 1. Hydraulic scheme of laboratory device for accelerated durability test

L_1, L_2, L_3, PEN – wires of three-phase electrical network; TR – transformer; C – cycler; RV – proportionally operated sequence pressure valve; P – pressure gauge; T – temperature sensor; HT – emergency button; M – electric motor; SO – rpm sensor; HG_1 – hydraulic pump (UD 25); F_1, F_2 – filters; ST – pressure sensor; HG_2 – hydrostatic pump of cooling circuit; CH_1, CH_2 – cooler; R – thermostatic regulator; V_1,V_2 – fans; N – tank; TV – two-stage sequence valve

operators do not clean up spills, thereby introducing pollutants to the environment. Using a fluid that is biodegradable reduces the cost of clean-up as well as the potential for polluting the environment (Cauffman et al. 2006). For this reason, there is an effort to replace the conventionally made mineral fluids with biodegradable fluids. Vegetable oils have a capability to contribute towards the goal of energy independence and security since they are a renewable resource (Campanella et al. 2010).

At present, technical means are at such a high level that there is space for the search and application of innovations that are based on environmental protection. This results in the development and production of lubricants that originate in renewable natural raw materials, either because of limited resources of fossil raw materials or environmental protection (Wagner et al. 2001). Before their first use in operation, fluids must be subjected to laboratory tests to prevent or minimize risk of damage of expensive agriculture machinery. At present, these fluids are underrepresented in agricultural machinery despite their low environmental impact and good operating and physico-chemical properties.

MATERIAL AND METHODS

The used fluid is a newly developing ecological fluid, which is made of synthetic fluid based on poly-alpha-olefins. It was chosen because it has high chemical stability and miscibility with mineral fluids currently used in tractors in Slovakia. During the test, a new ecological fluid MOL Farm UTTO Synt produced by MOL Group, Budapest, Hungary was used. This fluid belongs to the group of universal transmission hydraulic fluids designed for tractors. Because the biodegradable fluid is newly developed, its producer did not provide detailed specification, only the main parameters that are listed in Table 1.

The tested synthetic-based fluid was used in a laboratory test device that loaded the hydrostatic pump UD 25 (Jihostroj, Velešín, Czech Republic). The hydrostatic pump belongs to one-way hydrostatic pumps, which are used in the latest Zetor Forterra tractors for a common gear-hydraulic fill. The test device published by Hujo et al. (2012), Kosiba et al. (2013), Majdan et al. (2013) and Tkáč (2006) was designed at the Department of Transport and Handling, Faculty of Engineering, Slovak University of Agriculture in Nitra (Fig. 1).

Table 1. Specification of biodegradable synthetic-based hydraulic fluid MOL Farm UTTO Synt

Parameter	Value
Kinematic viscosity at 100°C (mm^2/s)	10.22
Kinematic viscosity at 40°C (mm^2/s)	58.14
Viscosity index (–)	165
Pour point (°C)	–42

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(a)

(b)

Fig. 2. Digital recording unit HMG 2020 (a) and sensor EVS 3100 (b)

In the design of laboratory device the main intention was to approach towards operating conditions as possible. For that reason, the hydraulic components, seals, filters and others that are contained in hydraulic circuit of agricultural tractors were selected as standard. Therefore, standard seals were used.

The principle of the test device operation is in loading the hydrostatic pump HG_1 (Jihostroj, Velešín, Czech Republic) by cyclic pressure load using an electro-hydraulic control valve RV, which is connected to the output of the hydrostatic pump. A change in the control valve position will change the direction of fluid flow, which then flows through the pressure relief valve TV into the tank T, or directly into the tank with fluid. These directional changes of flow result in pressure changes at the hydrostatic pump output. The hydrostatic pump is loaded with cyclic pressure load for the duration of 10^6 cycles, at rated parameters (Tkáč 2006). The hydraulic scheme of laboratory test device is in Fig. 1. During the laboratory fluid test, flow values are recorded in specified intervals (250,000 cycles) at rated parameters (pressure – 20 MPa, operating speed – 1,500 min^{-1}), using a digital recording unit HMG 2020 (HYQUIP Ltd., Horwich, United Kingdom) (Fig. 2a) and sensor EVS 3100 (Hydac, Sulzbach, Germany) (pressure sensor, flow sensor and temperature sensor in one) (Fig. 2b).

The measuring flow range of sensor EVS 3100 is from 6.0 to 60.0 dm^3/min, fluid temperature ranges from –20°C to 90°C, operating pressure is 40×10^6 Pa and accuracy is ≤ 2% of the actual value. The curves of flow characteristics of the used hydrostatic pump were created from measured results. After that, flow values were statistically processed by standardized normal distribution Eqs (1) and (2), and the flow efficiency Eq. (5) was calculated with modified formula Eq. (4) and then the decrease of flow efficiency Eq. (3). The limit value of flow efficiency decrease is 20%. This limit value is given by the manufacturer of the fluid.

Statistical method for the evaluation of fluid

Normal distribution. It says that continuous random variable x has normal (Gaussian) distribution with parameters μ, σ^2 if density is:

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} e^{-\frac{(x-\mu)^2}{2\sigma^2}} \quad \text{for } x \in R_1,\ \mu \in (-\infty, \infty),\ \sigma > 0 \quad (1)$$

where:
e – base of natural logarithm; σ – standard deviation of x; μ– mean of x

Standard normal distribution. The standard normal distribution is a special case of normal distribution. It is the distribution that occurs when a normal random variable has a mean of zero and a standard deviation of one. The normal random variable of standard normal distribution is called a standard score or a z-score. Every normal random variable X can be transformed into a z score via the following equation:

$$Z = \frac{x - \mu}{\sigma} \quad (2)$$

where:
σ – standard deviation of x; μ – mean of x; Z – standard score, z score

The variable has normal distribution with mean value 0 and variation 1 (then standard deviation is

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also 1). This distribution is called *standardized normal distribution* (Hill, Lewicki 2006).

Flow efficiency. The hydraulic fluid is evaluated based on the technical conditions of the hydrostatic pump, i.e. based on the loss of flow efficiency Eq. (3):

$$\Delta\eta_{pr} = \frac{\eta_{pr0} - \eta_{prm}}{\eta_{pr0}} \times 100 \qquad (3)$$

where:

$\Delta\eta_{pr}$ – loss of flow efficiency (%); η_{pr0} – flow efficiency at 0 cycles; η_{prm} – flow efficiency after 10^6 cycles

The calculation is based on consideration that the measured flow is equal to the flow calculated from theoretical flow and flow efficiency Eq. (4) (Petranský et al. 2004):

$$Q = Q_t \times \eta_{pr} = V_G \times n \times \eta_{pr} \qquad (4)$$

where:

η_{pr} – flow efficiency (%); Q – measured flow (dm^3/min); Q_t – theoretical flow (dm^3/min); V_G – geometrical volume (dm^3); n – speed of hydrostatic pump (min^{-1})

Flow efficiency is then given by Eq. (5):

$$\eta_{pr} = \frac{Q}{V_G \times n} \times 100 \qquad (5)$$

where:

V_G – geometrical volume (dm^3); n – speed of hydrostatic pump (min^{-1}); Q – measured flow (dm^3/min)

FTIR (Fourier Transform Infra Red) spectroscopy. The biodegradable fluid was studied by means of FTIR spectroscopy. In this method, light from the source which is passing through the fluid sample is divided into two beams by using a silver mirror. One of the beams is reflected from the fixed mounted mirror, and the other is reflected from the sliding mirror through which there is a time delay. The beams of coherent light source are measured at different settings of time delay. As they are measured at many positions of sliding mirror settings, the spectrum can be processed by the Fourier transformation from the temporal coherence of light (Fig. 3a).

Fig. 3b shows the FTIR spectrometer AVATAR 330 (LabX, Midland, Canada) and the results processed by the OMNIC software. Spectral range of spectrometer is from 7,800 to 375 cm^{-1}, optical resolution < 0.9 cm^{-1}, wavenumbers precision 0.01 cm^{-1} at 2,000 cm^{-1}.

ICP spectroscopy (Inductively Coupled Plasma) is an emission method to determine values of chemical elements by argon plasma. This method allows determination of all elements in the same series. Its disadvantage is in higher operating costs (Peťková 2012). The fluid sample is mixed with solution so that it can be pumped and is burned in a plasma torch, which is usually overheat by gas (argon) to about 13,000°C. The emitted radiation from the fluid sample during combustion is formed by radiation of each present element.

This spectrum is divided according to individual metals, and the amount of light is proportional to the amount of present metal. Particles up to 15 µm have no effect on the effectiveness of determination due to higher temperature during excitation of

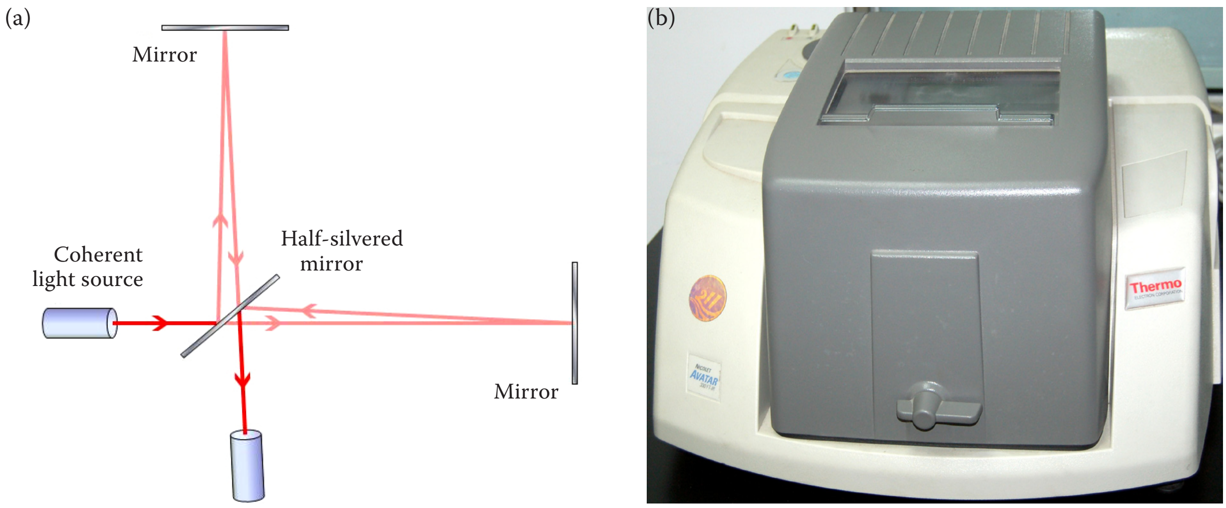

Fig. 3. Principle of FTIR spectroscopy (a) and spectrometer Avatar 330 (b)

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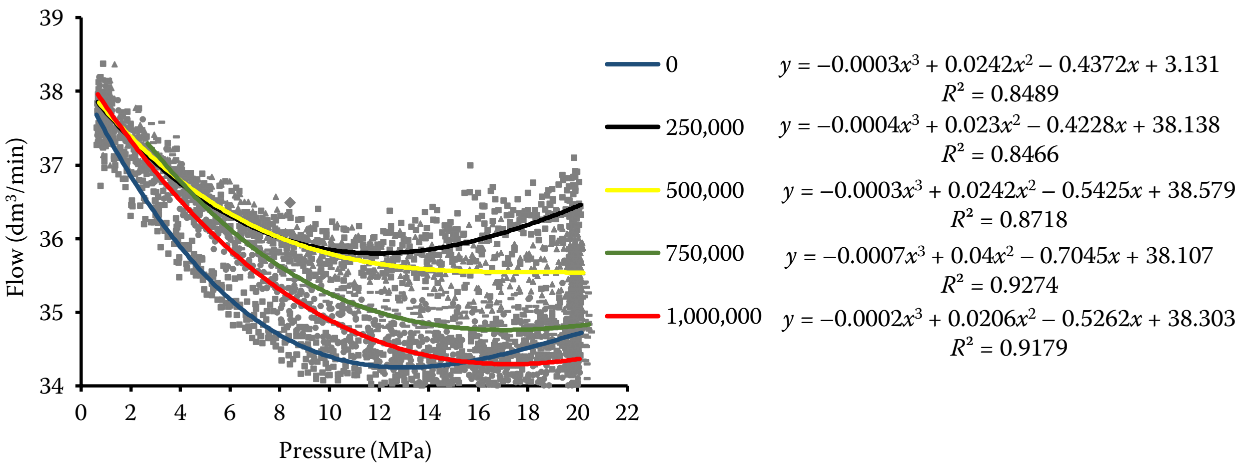

Fig. 4. Flow characteristics of hydrostatic pump UD 25

atoms. The limit value of particle size is 50 μm. The disadvantage of the direct method is that a sample is not completely homogeneous (Tkáč et al. 2007).

RESULTS AND DISCUSSION

The values of flows were obtained by the digital recording unit HMG 2020 every 250,000 cycles of pressure load.

The values of flow „*Q*" were statistically evaluated. The calculation is based on consideration that the measured flow is equal to the flow calculated from theoretical flow and flow efficiency Eq. (4). From the values of measured flows during test, the mean and standard deviation were calculated. Then, sample values were converted to standard score form „*Z*" on the basis of Eq. (2). The values of the sample of flows and their standardized values are shown in Table 2. Based on the selection of interval -1σ and $+1\sigma$, 68.27% of flow values were chosen. Then, flow efficiency Eq. (5) and the loss of flow efficiency Eq. (3) were calculated from the sample of flow values.

Subsequently, the flow values were processed and transferred in the form of flow characteristics (Fig. 4). Fig. 4a shows the courses of flow characteristics measured every 250,000 cycles. It can be seen that the hydrostatic pump UD 25 improved its operational parameters during the test; higher values of flows were measured at nominal pressure 20 MPa, compared to the measured flow values at the beginning of the test (new hydrostatic pump). After completion of the test (1,000,000 pressure load cycles), the hydrostatic pump showed a decrease of flow. To better illustrate the flow courses and size of change in technical conditions, the technical conditions of the hydrostatic pump will be expressed by a decrease of flow efficiency (Fig. 5).

Based on the results of flow efficiency decrease, it can be stated that the hydrostatic pump UD 25 improved its operational parameters up to 750,000 cycles, which is characterized by negative values of decrease of flow efficiency. The negative value of flow efficiency is attributed to the running-in process. The highest negative value was recorded at 250,000 cycles, i.e. –4.90%. Then the hydrostatic pump reached the best operational parameters and thus the highest flow. During the laboratory test, there was a gradual deterioration of operational parameters, and at the end of test (1,000,000 cycles), a 1.03% decrease of flow efficiency was observed.

In this case, wear of the hydrostatic pump occurred in the initial phase of operation. On the basis of the measured values of flow efficiency decrease during the laboratory test, it can be stated that the

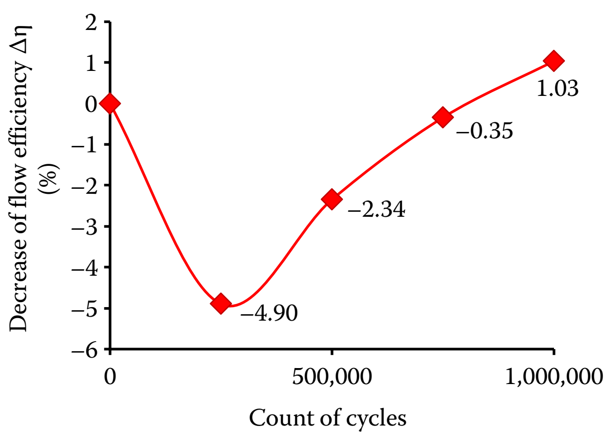

Fig. 5. Decrease of flow efficiency of hydrostatic pump UD 25

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Table 2. Statistically processed flow values of new hydrostatic pump UD 25 and hydrostatic pump at the end of test

New hydrostatic pump			Hydrostatic pump at the end of test		
N	*Q* (dm^3/min)	*Z*	*N*	*Q* (dm^3/min)	*Z*
1	34.77	2.28	1	34.94	1.93
2	34.83	2.05	2	34.18	1.93
3	34.74	2.05	3	34.25	1.78
4	35.13	2.05	4	33.83	1.56
5	34.93	2.05	5	34.29	1.34
6	34.41	1.70	6	34.1	1.34
7	34.55	1.70	7	34.22	1.2
8	35.00	1.35	8	34.22	1.2
9	34.93	1.12	9	34.05	1.2
10	34.73	1.12	10	34.25	1.2
11	34.34	0.78	11	34.55	1.2
12	34.48	0.78	12	33.84	0.98
13	34.72	0.78	13	34,32	0.98
14	34.42	0.78	14	34,65	0.98
15	34.61	0.78	15	34,42	0.98
16	34.68	0.92	16	33.78	0.76
17	34.67	0.92	17	34.7	0.76
18	34.58	0.92	18	34.28	0.76
19	34.67	0.92	19	34.54	0.76
20	35.06	0.93	20	34.52	0.62
21	34.48	0.92	21	34.2	0.61
22	34.67	0.92	22	34.73	0.62
23	35.44	0.94	23	34.36	0.62
24	34.03	0.91	24	34.02	0.39
25	35.00	0.93	25	34.45	0.39
26	34.55	0.92	26	34.28	0.39
27	35.43	0.94	27	34.75	0.28
28	34.04	0.91	28	34.65	0.28
29	34.42	0.92	29	34.74	0.28
30	34.78	0.93	30	34.04	0.28
31	34.62	0.20	31	34.53	0.28
32	34.62	0.20	32	34.99	0.28
33	34.47	0.20	33	34.68	0.28
34	34.55	0.20	34	34.43	0.28
35	35.06	–0.14	35	33.88	0.3
36	34.02	–0.24	36	33.70	0.03
37	34.42	–0.14	37	34.24	0.03
38	35.06	–0.14	38	34.53	0.03
39	34.57	–0.14	39	34.28	0.03

Table 2 to be continued

New hydrostatic pump			Hydrostatic pump at the end of test		
N	*Q* (dm^3/min)	*Z*	*N*	*Q* (dm^3/min)	*Z*
40	34.72	–0.14	40	34.33	–0.19
41	34.65	–0.14	41	34.28	–0.19
42	34.98	–0.14	42	34.00	–0.19
43	34.99	–0.49	43	34.42	–0.19
44	34.72	–0.49	44	34.50	–0.41
45	34.48	–0.49	45	33.84	–0.41
46	34.40	–0.49	46	34.39	–0.41
47	34.55	–0.49	47	34.88	–0.41
48	35.57	–0.49	48	34.48	–0.55
49	35.06	–0.72	49	33.93	–0.55
50	34.78	–0.72	50	34.27	–0.55
51	35.24	–0.72	51	33.78	–0.55
52	34.73	–0.72	52	33.53	–0.77
53	35.23	–0.72	53	34.72	–0.77
54	33.95	–0.72	54	33.82	–0.77
55	34.62	–1.07	55	34.25	–0.77
56	34.80	–1.07	56	35.24	–0.92
57	34.55	–1.07	57	34.35	–0.92
58	34.48	–1.07	58	33.80	–1.14
59	34.66	–1.07	59	34.32	–1.14
60	34.71	–1.41	60	34.45	–1.36
61	34.29	–1.41	61	34.41	–1.36
62	34.48	–1.41	62	34.30	–1.36
63	34.35	–1.41	63	34.00	–1.36
64	34.87	–1.41	64	34.55	–1.50
65	34.81	–1.41	65	34.20	–1.50
66	34.73	–1.41	66	33.41	–1.94
67	34.23	–1.41	67	34.91	–1.94
68	34.59	–1.41	68	34.26	–1.94
69	34.15	–1.41	69	34.42	–1.94

N – ranking number; *Q* – flow values; *Z* – statistical processed values of flow with standardized normal distribution according Eq. (2); the flow values with interval –1σ and +1σ are in grey area

hydrostatic pump had very good operational parameters when it was working with the newly developed synthetic biodegradable fluid because the resulting value of flow efficiency decrease did not exceed the limit value 20% specified by the manufacturer (Majdan et al. 2013; Máchal et al. 2013).

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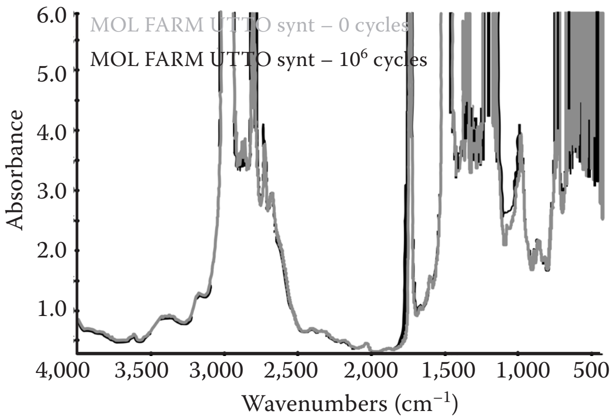

Fig. 6. Whole IR spectrum of synthetic biodegradable fluid MOL Farm UTTO Synt at the beginning of test and at the end of test

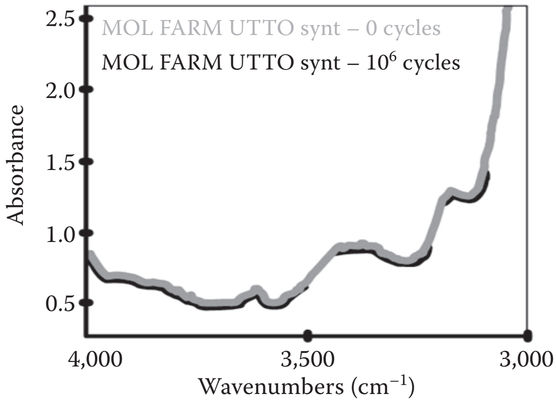

Fig. 7. Peaks overlap of evaluation of thermal oxidation and water content of new biodegradable fluid

During the accelerated laboratory test worsening of technical state of seals used in hydraulic circuit of laboratory device was not recorded.

FTIR spectroscopy

In Fig. 6, the IR spectra of new biodegradable fluid before the start and after the end of laboratory test are compared. The evaluation of whole IR spectrum was done on the basis of changes (increase or decrease of peaks) monitored during the courses of individual spectra (Peťková 2012).

In the area of 3,540 cm^{-1} wawenumbers, the oxidation of fluid based on overheating (Fig. 7) was evaluated. Temperature in the laboratory device was kept at the specified value, which was determined by operational measurements on the tractor. In terms of oxidation due to temperature, the fluid shows good properties and is suitable for the given tractor. In the area of 3,625 cm^{-1} wawenumbers, it is possible to monitor if there is water in fluid. The lines and peaks overlap, therefore it is not possible to see some (increase or decrease) changes of peaks in this area (Fig. 8a) only negligible changes can be noticed.

Detergents are characteristic in the area of 1,630 cm^{-1} (Fig. 8b). Since there was no change of peak in this area, depletion of additive did not occur during the test and the fluid maintained good quality properties. In the areas of 880 cm^{-1} and 1,040 cm^{-1}, oxidation products based on formation of acid components in the fluid are monitored (Fig. 9). There is a small increase of peaks, indicating the initial phase of fluid degradation.

The area of 980 cm^{-1} is characteristic for ZDDP-based additives and corrosion inhibitors. No changes were recorded in this area.

Evaluation of additives on the basis of the ICP spectroscopy

In case of the ICP spectroscopy, decrease or increase of individual chemical elements is evaluated and based on the results, it is possible to monitor the operational parameters of fluids (Peťková 2012). The amount of chemical elements enables to measure additives in fluid, the presence of which is shown in the form of increasing and decreasing levels of specific chemical elements (Ca, P, Zn and Mg).

The presence of increased zinc (Zn) value points out to additives on the basis of zinc dialyldithiophosphate (ZDDP). This category contains antiwear additives, high-pressure additives, antioxidants and demulsifiers. The increased presence of chemical elements calcium (Ca) and magnesium (Mg) in the results of the ICP spectroscopy indicates the presence of corrosion inhibitors and partly detergents. The increased level of chemical element phosphorus (P) indicates the presence of metal deactivators, retardants of ageing and antioxidants.

Based on the results of the ICP spectroscopy (Fig. 9), it can be stated that during the laboratory test, there was no sudden decrease in the observed chemical elements and thus the depletion of additives contained in the synthetic biodegradable fluid. During the whole time of accelerated durability

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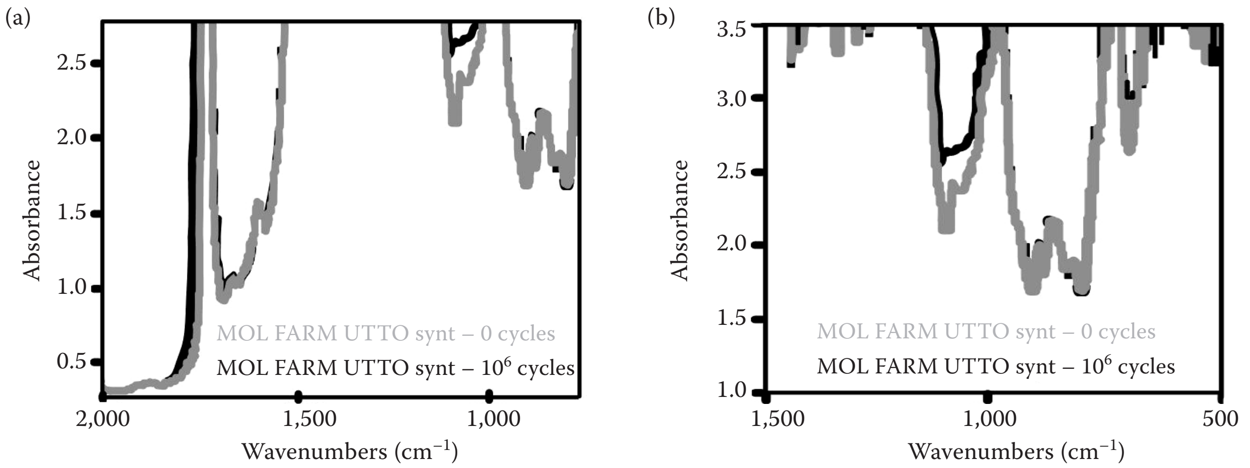

Fig. 8. Peaks change in (a) areas of acid impact products and areas of detergents and (b) in area of acidic oxidation by acid products and area with no change of additives on the basis of ZDDP and corrosion inhibitors

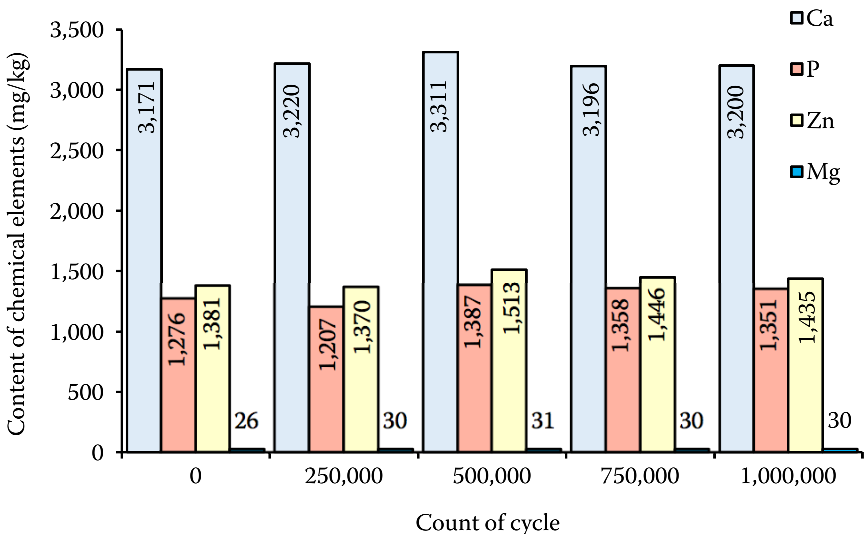

Fig. 9. Contents of selected chemical elements in synthetic biodegradable fluid

test of the hydrostatic pump, the fluid maintained good properties in terms of depletion of additives.

CONCLUSION

During the laboratory test, the newly developed synthetic biodegradable fluid MOL Farm UTTO Synth, was evaluated; it belongs to the group of gear-hydraulic fluids. The fluid was evaluated based on the results from laboratory durability test of a hydrostatic pump UD 25, which is used in the new tractors Zetor Forterra.

During the test, the hydrostatic pump was in the running-in process, which is characterized by the negative value of decrease of flow efficiency. At the end of the test, a 1.03% decrease of flow efficiency was recorded. In view of impact on the technical conditions of the hydrostatic pump, the fluid demonstrated very good properties because the limit value specified by the manufacturer (20%) was not exceeded. The ICP spectroscopy was used to evaluate the presence of specified chemical elements, the increasing or decreasing value of which characterizes the presence of certain types of additives. In view of additives depletion by ICP spectroscopy, it can be stated that there was no excessive additives depletion as confirmed by the results of FTIR spectroscopy that show increases of peaks recorded in the areas 880 cm^{-1} and 1,040 cm^{-1}, indicating the initial phase of fluid degradation in terms of formation of acid components. Water content and overheat-

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ing degradation were in normal state. As was mentioned above, the European Union with tightening legislation focuses on the environment protection and requires gradual replacement of conventionally produced fluids by biodegradable fluid.

The impact of these fluids on hydraulic circuit of agricultural machine has not been fully researched yet, and therefore it is necessary to dedicate research to biodegradable fluids and their impact on technical state of parts of hydraulic system. On the basis of the test results of new biodegradable fluid MOL Farm UTTO Synt it is possible to affirm that the fluid may potentially replace conventionally made mineral fluids and is suitable for subsequent test under operational conditions without potential damage of expensive agricultural machinery and thus it contributes to environmental protection.

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