ARISEN

Innovation in Lubrication for Sustainable Manufacturing

Monitoring Meeting

Online, 11th May 2023

Agenda

01

Introduction of the participants

02

Project Presentation

Introduction of the participants

Turkey	Portugal	
BELGIN Developing of the lubricant, productisation, exploitation Established 1953	**Centimfe** Testing, standardization, dissemination Established 1991	**Toolpresse** End-use validation of the lubricant Established 2001

ARISEN
innovAtion in lubRicatIon for SustainablE maNufacturing

Project Duration	36 months
Total Effort (PY)	161,2
Total Budget (k€)	979,67
Project Coordinator	BELGIN

Reasons for initiating the project

- ✓ The demand for the use of aluminum and titanium alloys in the industries is increasing.
- ✓ Need to develop of more economical and environmentally friendly production technologies that will replace the traditional flood cooling approach
- ✓ Need to produce alternative lubricants that can ensure sustainability due to the depletion of petroleum reserves.
- ✓ With the increasing demands for both **sustainability** and **green manufacturing**, there is a need to develop MQL technologies

- ✓ Despite recent attempts to use vegetable-based lubricants, no real alternative has yet been found.
- ✓ Project studies have been started in order to introduce an innovative and value-added product to the market in accordance with future market trends and needs.

The objective of the project

The aim of the project is to meet the demands of the global market for the processing of Al 6000 and 7000 series and Ti6Al4V light alloys, and to develop a **sustainable, environmentally friendly**, **biodegradable** product **suitable for the MQL system**.

Project innovations and technology value chain

The biggest and **most important difference** and **innovative aspect** of ARISEN from the literature and existing studies is that it offers not only a product but also a solution package, namely a concept. This concept will include 4 key elements.

1. it is aimed to develop a lubricant that can be used in both titanium and aluminum processing.
2. The product to be developed will make the use of the MQL system attractive despite the high investment cost with its performance.
3. Air cooling in the MQL system will be removed.
4. The effects of different machining parameters will also be determined, thus creating a new business model.

R&D goal for ARISEN

With the ARISEN project,

- ✓ To develop a lubricant that can be used in both titanium and aluminum machining.
- ✓ A new lubricant for the MQL system
- ✓ Air cooling will be removed in the MQL system.
- ✓ Biodegradable product.
- ✓ A new business model will be created.

Impact

The most important effect and strength of the project is that it is based on the cooperation of 3 expert companies.

So ARISEN has the ambition to cover and impact the whole value chain.

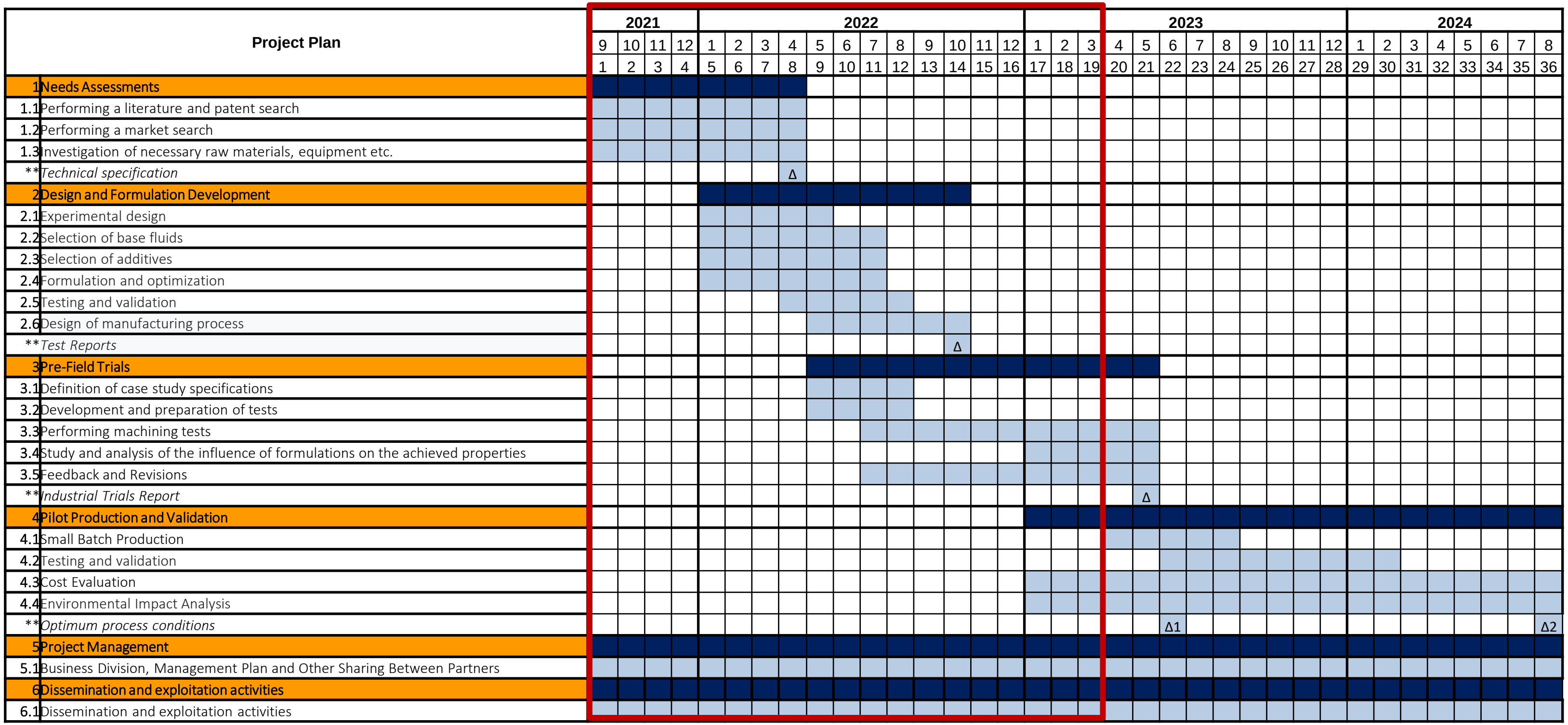
- Manufacturing process
- Bio-lubricants production
- MQL system manufacturers
- Lubricant manufacturers
- Manufacturing industry

Expected Project Outputs

ARISEN will lead to three core outputs:

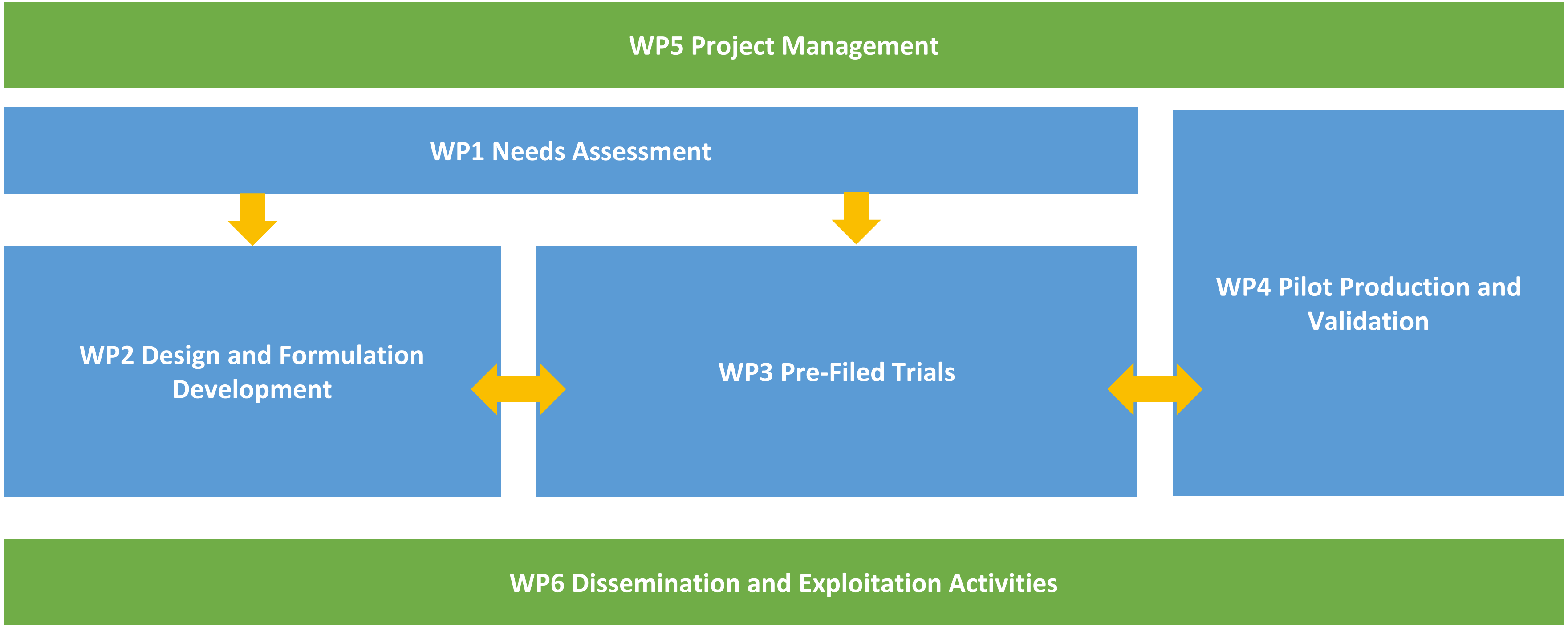
- O1: A product line of mineral-oil free and biodegradable lubricants for the metal industry
- O2: MQL machining of titanium and aluminum alloys by means of the ARISEN lubricants
- O3: Sustainability in the manufacturing processes in the metal industry where ARISEN lubricants are deployed

Gantt Chart



Project Plan
2021
2022
2023
2024
9 10 11 12 1 2 3 4 5 6 7 8 9 10 11 12 1 2 3 4 5 6 7 8 9 10 11 12 1 2 3 4 5 6 7 8
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36
1 Needs Assessments
1.1 Performing a literature and patent search
1.2 Performing a market search
1.3 Investigation of necessary raw materials, equipment etc.
** Technical specification
Δ
2 Design and Formulation Development
2.1 Experimental design
2.2 Selection of base fluids
2.3 Selection of additives
2.4 Formulation and optimization
2.5 Testing and validation
2.6 Design of manufacturing process
** Test Reports
Δ
3 Pre-Field Trials
3.1 Definition of case study specifications
3.2 Development and preparation of tests
3.3 Performing machining tests
3.4 Study and analysis of the influence of formulations on the achieved properties
3.5 Feedback and Revisions
** Industrial Trials Report
Δ
4 Pilot Production and Validation
4.1 Small Batch Production
4.2 Testing and validation
4.3 Cost Evaluation
4.4 Environmental Impact Analysis
** Optimum process conditions
Δ1
Δ2
5 Project Management
5.1 Business Division, Management Plan and Other Sharing Between Partners
6 Dissemination and exploitation activities
6.1 Dissemination and exploitation activities

Work Packages Flow



WP1: Need Assessments

	Project Plan	2021				2022															2023									2024							
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		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
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1.3	Investigation of necessary raw materials, equipment etc.																																				
**	*Technical specification*								Δ																												

Task 1.1 : Performing a literature and patent research (Task Leader: Belgin, Task Participants: Centimfe and Toolpresse) M1-M8

Task 1.2 : Performing a market search (Task Leader: Centimfe, Task Participants: Belgin and Toolpresse)) M1-M8

Task 1.3 : Investigation of necessary raw materials, equipment etc. (Task Leader: Belgin, Task Participants: Centimfe) M1-M8

WP1: Need Assessments

Establishing the planning in the project is the most important part and all three partners contributed to the work package that includes this needs analysis.

- ✓ Literature Search
- ✓ Market Search
- ✓ Raw Materials

WP1: Need Assessments

Performing a Literature and Patent Search

Within the scope of literature research, article, compilation, book and patent research activities were carried out in order to increase learning and provide knowledge.

Publication name	Year	Author(s)	Summary information that will provide input to the project:
Development of ecofriendly/biodegradable lubricants: An overview	2012	P. Nagendramma, S. Kaul	This review provides evidence for the rapid increase in the use of biodegradable products in the field of metalworking fluids and the increasing use of biodegradable synthetic esters in this field.
Milling and Turning of Titanium Aluminides by Using Minimum Quantity Lubrication	2014	Priarone, P. C., Robiglio, M., Settineri, L., & Tebaldo, V.	Information has been gained about the harsh machining conditions of materials such as titanium and the disadvantages of using conventional cutting fluids.
An experimental investigation of effect of minimum quantity lubrication in machining 6082 aluminum alloy	2014	R. Yiğit	It has been learned that MQL improves its environmental friendliness (regular protection, providing a clean and dry working environment, preventing discomfort and health hazards caused by heat, smoke, gases, etc., and preventing environmental pollution) and machinability properties.
A Critical review on Minimum Quantity Lubrication (MQL) Coolant System for Machining Operations	2016	Madhukar S., Shravan A., Vidyanand P., Reddy G. S.	Important information was obtained about the different MQL systems, the duties of cooling oils and coolant systems, and the advantages of using vegetable-based lubricants in MQL systems.
Investigation of the Effect of Minimum Quantity Lubrication (MQL) on the Machining of Titanium and its Alloys A Review	2017	CAGAN S. Ç. ve Buldum B. B.	Gained knowledge on the machinability of titanium under MQL.
Eco-Friendly Cutting Fluids in Minimum Quantity Lubrication Assisted Machining: A Review on the Perception of Sustainable Manufacturing	2021	Sen B., Mia M., Krolczyk G. M., Mandal U. K · Mondal S. P.	Deep knowledge about the concept of sustainability and the applicability of this concept in MQL systems has been obtained.
Metalworking fluids	2006	SUDA S., Yokota H., Masanori I	Metalworking oil has been reported to be suitable for cutting, grinding and roll forming with a minimal amount of lubrication system.

WP1: Need Assessments

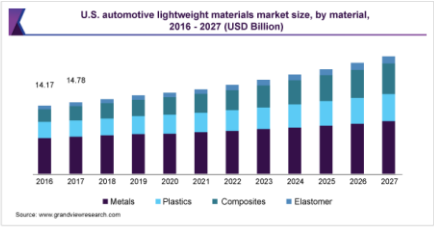
Performing a Market Search

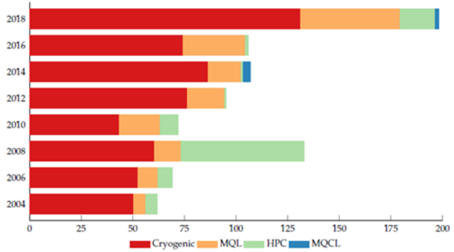
Market search was conducted in order to analyze the needs of the sector and to understand what needs and how much the project output could meet.

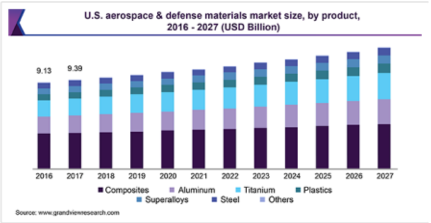
- ✓ Global Aluminum and Titanium market
- ✓ Aluminum and Titanium machining sectors
- ✓ Current and Sustainable machining techniques and technologies
- ✓ Metalworking fluids market

We have evolved our work into research on ;

- ✓ Machining technologies
- ✓ Current solution technologies.







WP1: Need Assessments

<u>Investigation of necessary raw materials, equipment etc</u>

<u>Raw materials</u>

- ✓ Consideration of parameters such as viscosity, thermal and chemical stability, percent biodegradation, lubricant performance, additive compatibility
- ✓ Additives, that will not leave any stains on the Al
- ✓ Chlorine-free additives
- ✓ Compliance with REACH (Registration Assessment Authorization and Restriction of Chemicals) regulation.
- ✓ Supply of TDS, SDS and samples

WP1: Need Assessments

Investigation of necessary raw materials, equipment etc

Equipment etc

- ✓ Tribological test devices
- ✓ Al and Ti test plates and test taps for Microtap
- ✓ Availability of Al 6061-7075 and TiAl6V4 alloy test materials for use for SRV
- ✓ Usage of Profilometer (3D Microscope)
- ✓ Research and supply of machines, cutting tools, aluminum and titanium materials to be machined, measuring equipment and MQL system.
- ✓ Performing the OECD 301B test
- ✓ Performing the oil mist test
- ✓ Using the Design Expert program

WP1: Need Assessments

Investigation of necessary raw materials, equipment etc

- ✓ Supply and inspection of 2 current solution products

Features	SAMPLE-1 (O)	SAMPLE-2 (C)	Test Method
Appearance	Light Yellow, Clear	Amber, Clear	-
Emulsion Appearance (%5)	Translucent	Translucent	-
Kinematic Viscosity (40°C, cSt)	57,51	55,16	ASTM D 445
Pour Point (°C)	-3	0	ASTM D 97
Flash Point (°C)	>100	>100	ASTM D 92
Foam Test (ml/ml)	250/0	200/0	BLN 35
pH (%5, 10 °dH)	9,05	8,96	ASTM D 1287
SRV EP Step Test (N) (Al/Ti)	300	400	ASTM D 5706
Tapping Torque Test, Al/Ti (Nm)	166,9 (Ti) (Al 6061): 102,2 (Al 7075): 155.2	Uygulanamaz (Ti) (Al 6061): 122,23 (Al 7075): 192,58	BLN 105
RVT Wear Test (mm²)	16,96	26,69	-
Corrosion Test (10 °dH, %5, 2 h)	0-0	0-0	DIN 51360/2
Corrosion Test (10 °dH, %3, 2 h)	1-0	2-3	DIN 51360/2
Refractometer Factor	1,28	1,06	Hand Refractometer
Cleanability Test	Easy	Easy	BLN 147
Biodegradability Test	Not determined		OECD 301

WP1: Need Assessments

**Creation of Technical Specification

✓ As a result of the preliminary studies and meetings held with Centimfe and Toolpresse, Belgin Product Technical Specification and Centimfe-Toolpresse Industrial Trial Technical Specification were created for the final formulation.

Product Technical Specification

Technicial Specifications	Limit Values	Test Method
Kinematic Viscosity (40°C, cSt)	10-500	ASTM D 445
Pour Point (°C)	max -10	ASTM D 97
Flash Point (°C)	min 140	ASTM D 92
Total Acid Number (TAN) (mg KOH/g)	max 5,0	ASTM D 974
Foaming Tendency and Stability (mL/mL, Kd. I)	max 50/0	ASTM D 892
SRV Friction Test (fmax) (Al/Ti)	max 0,500	ASTM D 6425
SRV EP Step Test (N) (Al/Ti)	min 400	ASTM D 5706
Tapping Torque Test Al/Ti (Nm)	max 350	BLN 105
Rust Test	Pass	ASTM D 665 A
Aluminum Stain Test	Pass	BLN 99
Wettability Test	Good	BLN 172
Cleanability Test	Should be easy	BLN 147
Biodegradability Test	min %80	OECD 301

WP1: Need Assessments

** Creation of Technical Specification

Field Technical Specification

Turning and milling operations will be applied.

- Aluminum turning: polished carbide inserts, titanium turning: TiAlN coated carbide inserts
- Aluminum milling: integrated carbide cutting tools, titanium milling: TiAlN coated carbide tools
- Milling of aluminum and titanium: Integrated carbide cutting tools with a diameter of 10 mm, 4 flutes and a corner radius of 2.5 mm,
- Aluminum and titanium turning: 55º and 0.2mm corner radius carbide inserts,
- External MQL system will be used.

Trials will be carried out under the following operating conditions.

Technicial Specifications	Aluminum turning	Titanium turning	Aluminum milling	Titanium milling
Two levels of cutting speed (Vc) (m/min)	250- 400	50-80	250-400	50-80
feed per revolution (fn) in two levels (fn) (mm/rev)	0.07- 0.15	0.05-0.1	0.06-0.12	0.05-0.1
Two levels of (axial) depth of cut (ap) (mm)	0.2- 0.3	0.05-0.2	0.05-0.1	0.05-0.1
MQL spray amount in two levels (ml/h)	40- 80	60-150	40-80	60-150

WP2: Design and Formulation Development

Project Plan		2021				2022															2023									2024							
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2.6	Design of manufacturing process																																				
**	*Test Reports*														Δ																						

Task 2.1: Experimental design (Task leader: Belgin) M5-M9

Task 2.2: Selection of base fluids (Task leader: Belgin) M5-M11

Task 2.3: Selection of additives (Task leader: Belgin) M5-M11

Task 2.4: Formulation and optimization (Task leader: Belgin) M5-M11

Task 2.5: Testing and validation (Task leader: Belgin) M8-M12

Task 2.6: Design of manufacturing process (Task leader: Centimfe, Task participants:Belgin and Toolpresse) M9-M14

WP2: Design and Formulation Development

Experimental design :

✓ Purpose of experimental design and experimental design studies

FORMULATION TRIALS

		Base Fluid Trials 1					Surfactant Trials 2					Anti-Wear Trials 3							Antioxidant Trials 4						Corrosion Inhibitor Trials 5						Anti-Foam Trials 6					
Sample Code																																				
Number of Samples		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35
1	Amount of Additives % g/g	X1																																		
2			Y1																																	
3				Z1																																
4					A1		A1	A1	A1	A1	A1	A1																								
5						B1																														
6							S1				S1																									
7								S2		S2	S2	S2																								
8									S3	S3			T2	T2	T2	T3	T4	T5	T6	T7	T8	T9	T10	T11	T12	T13	T14	T15	T16	T17	T18	T19	T20	T21	T22	T23
9												XY1			XY2	XY3	XY1		XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3
10													XY1			XY3	XY2	XY4	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3	XY3
11														XY1	XY2		XY3	XY4																		
12																			OK1		OK12		OK15	OK16	OK12	OK12	OK12	OK12	OK12	OK12	OK12	OK12	OK12	OK12	OK12	OK12
13																				OK2	OK13	OK14		OK17	OK13	OK13	OK13	OK13	OK13	OK13	OK13	OK13	OK13	OK13	OK13	OK13
14																									KY1		KY4		KY7	KY8						
15																										KY2	KY5	KY6		KY9	KY2	KY2	KY2	KY2	KY2	KY2
16																															F1		F3		F6	F7
17																																F2	F4	F5		F8
IR	TESTS																																			
Pour Point																																				
Flash Point																																				
Viscosity 40 ℃																																				
Refractive Index																																				
Stability																																				
SRV Disc Aw																																				
SRV EP																																				
F max																																				
4 Ball AW																																				
X RAY																																				
Rust Test																																				
RBOT																																				
Foam Test																																				

WP2: Design and Formulation Development

Selection of Oil:

Five types of biodegradable oil structures have been investigated:

- ✓ Vegetable oils, low viscosity polyalphaolefins (PAOs), polyalkylene glycols (PAGs), dibasic acid esters (DEs) and polyol esters such as neopentyl glycol dioleate (NPG-DO), trimethylolpropane trioleate (TMP-TO), trimethylolpropane complex ester (TMP-CX) pentaerythritol tetraoleate (PE-TO).
- ✓ The use of vegetable oil and esters in formulation studies,
- ✓ Metalworking performances of canola and castor oils and polyol esters obtained from these oils on Aluminum and Titanium test pieces,

Features	Test Method	Castor oil	Canola Oil	NPG-DO	TMP-TO	TMP-CX	PE-TO	Limit Values
Kinematic Viscosity (40°C, cSt)	ASTM D 445	150	46	23	46	64	65	10-500
Pour Point (°C)	ASTM D 97	≤-21	≤-18	≤-18	≤-42	≤-33	≤-25	max -10
Flash Point (°C)	ASTM D 92	172	≥ 190	≥ 250	≥ 270	≥ 270	≥ 280	min 140
Total Acid Number (TAN) (mg KOH/g)	ASTM D 974	≤ 2	≤ 2	≤ 1	≤ 1	≤ 1	≤ 1	max 5,0
SRV EP Step Test (N) (Al/Ti)	ASTM D 5706	300	500	500	500	400	500	min 400
Tapping Torque Test Al 6061 (800 rpm, d 14,4mm) (Nm)	BLN 105	164,35	144,57	146.13	140.20	142.51	137.32	max 350
Tapping Torque Test Al 7075 (800 rpm, d 14,4mm) (Nm)	BLN 105	254,2	234.7	240.7	226.8	233.9	225.9	Max 350
Tapping Torque Test Ti-6Al-4V (300 rpm, d 5mm) (Nm)	BLN 105	149,9	152.8	187.1	146,5	147.4	148.2	max 350
Biodegradability Test	OECD 301	100 %	100 %	96 %	100 %	100 %	76 %	min %80

<u>**As a result of these tests, it was decided to carry out formulation studies with ester in TMP-TO structure, which has stable machining performance for all alloys.**</u>

WP2: Design and Formulation Development

Selection of Oil:

Alternative to the work of polyol esters;

- ✓ Lubricant trials with next-generation polymeric esters based on renewable resources from oleic acid and sugar cane oil
- ✓ Hydrolytic and thermal stability
- ✓ Metalworking performances on Aluminum and Titanium test pieces.

Features	Test Method	PE-1	PE-2
Kinematic Viscosity (40°C, cSt)	ASTM D 445	465	3000
Pour Point (°C)	ASTM D 97	-12	18
Flash Point (°C)	ASTM D 92	150	204
Total Acid Number (TAN) (mg KOH/g)	ASTM D 974	50	20
SRV EP Step Test (N) (Al/Ti)	ASTM D 5706	400	700
Tapping Torque Test Al 6061 (800 rpm, d 14,4mm) (Nm)	BLN 105	146,5	108,3
Tapping Torque Test Al 7075 (800 rpm, d 14,4mm) (Nm)	BLN 105	233,0	169,3
Tapping Torque Test Ti-6Al-4V (300 rpm, d 5mm) (Nm)	BLN 105	148,0	146,2
Biodegradability Test	OECD 301	>80	>80

The use of these two polymeric esters was also evaluated with the project partners for their similar-good (PE-1) or even very good (PE-2) machining performance on all metal alloys. It was decided to carry out formulation studies with these two polymeric esters.

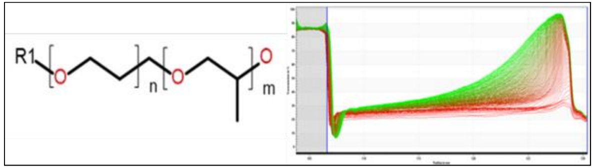
WP2: Design and Formulation Development

Selection of Additives:

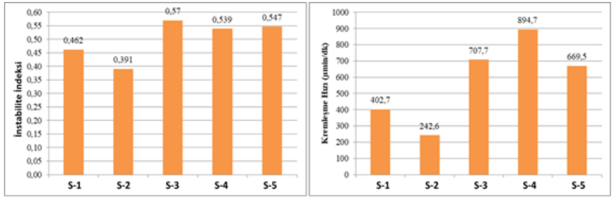
Evaluation of Emulsifier Additives:

- ✓ The use of ethoxylated/propoxylated fatty alcohols, ethoxylated/ propoxylated fatty acids, ethoxylated castor oils, ethoxylated/propoxylated glycerol and sorbitan esters and other surfactants,
- ✓ Tested with the Lumisizer.
- ✓ Easily biodegradable (301B (28 d): > 60 %) surfactant with Alkyl Polyglycol ether structure.
- ✓ Soybean oil and TMP type polyol ester with derivatives of this surfactant with different HLB values.
- ✓ Emulsifiers with high HLB value

LUMiSizer Test Machine



Surfactant basic molecular structure and delivery profile of formulations



Instability Index test charts of Formulations and Creaming speed test charts of Formulations

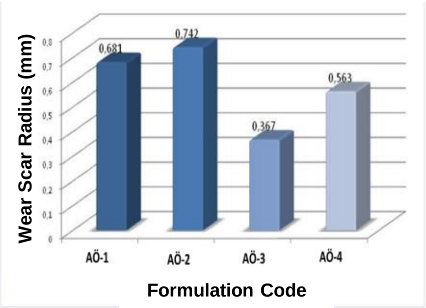
WP2: Design and Formulation Development

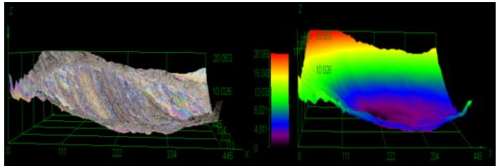
Selection of Additives:

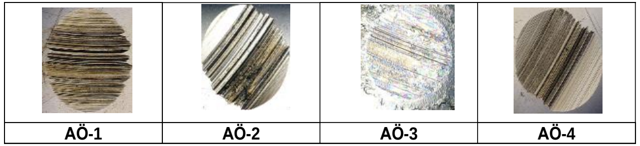
Evaluation of Anti-Wear Additives:

- ✓ The use of phosphorus-containing additives as anti-wear and/or extreme pressure additives,
- ✓ Phosphorus based additives such as phosphonates, phosphoric acids, organic phosphorus derivatives and zinc alkydithiophosphate etc.
- ✓ Comparison of anti-wear performances of phosphorus derivative additives with different chemical structures in TMP-TO formulations,
- ✓ The wear scar diameters and the volumes of the balls were examined under a 3D microscope,

It has been determined that the best anti-wear performance is provided by the phosphate ester derivative.





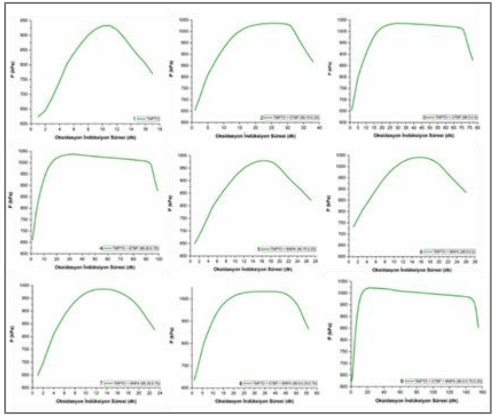


WP2: Design and Formulation Development

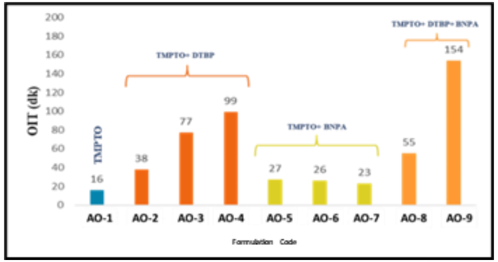
Selection of Additives:

<u>Evaluation of Antioxidant Additives:</u>

- ✓ 2,6-di-tert-butyl phenol antioxidants, and bis(nonylphenyl)amine antioxidant,
- ✓ The effect of the selected additives was determined by performing the RBOT oxidation test.
- ✓ The use of DTBP in minimum, medium and maximum ratios on TMP ester increases the oxidation resistance proportionally from minimum to maximum.
- ✓ Synergistic effect of these two antioxidant compounds



Oxidation induction time/pressure graph



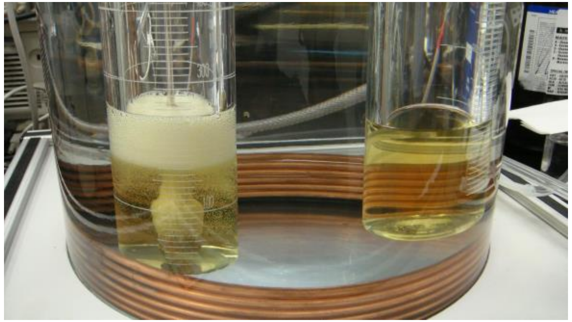
Numerical data graphs of oxidation induction time

WP2: Design and Formulation Development

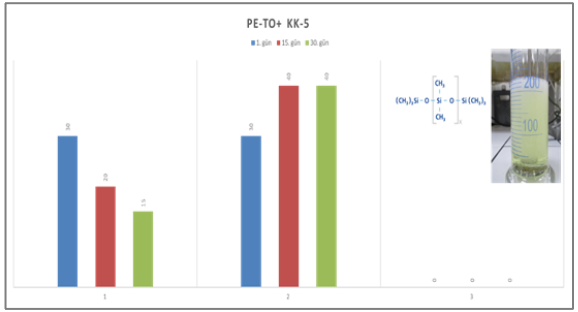
Selection of Additives:

Evaluation of Antifoam Additives:

- ✓ The importance of bubbling in MQL application,
- ✓ Defoamers suitable for use in biodegradable oils, such as acrylic acid, methacrylic acid or their hydroxyalkyl esters,
- ✓ Foam Test according to ASTM D 892 test norm,
- ✓ The best antifoam performance is with antifoam agents containing siloxane.



Foam test



Foam stability test graphics and test image applied to the formulation formed with siloxane derivative antifoam agents at the end of the 1st, 15th and 30th days.

WP2: Design and Formulation Development

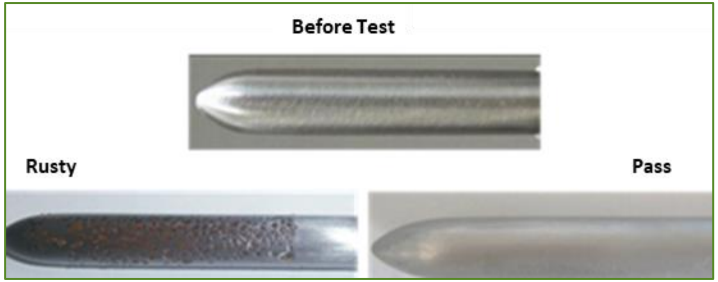
Selection of Additives:

Evaluation of Corrosion Inhibitors:

- ✓ Use of nitrogen-based additives such as imidazole, benzimidazole, 2-mercaptobenzothiazole, 2,5-mercapto-triazole and benzotriazole as metal deactivators
- ✓ Investigation of the performance effect of metal deactivator additives by Corrosion-Rust test
- ✓ Rust test according to ASTM D 665 A norm. (60°C, 4 hours, 30 ml distilled water)

Formulation Code	Rust Test
CI-1	PASS
CI-3	PASS
CI-4	PASS
CI-5	PASS
CI-7	PASS
CI-8	PASS
CI-10	PASS
CI-2	RUSTY/ NOT PASS
CI-9	RUSTY/ NOT PASS
CI-6	RUSTY/ NOT PASS

Rust test results of corrosion inhibitors.



WP2: Design and Formulation Development

Formulation and optimization

OPTIMIZATION TRIALS													
		Anti-Wear			Antioxidant			Corrosion Inhibitor			Anti-Foam		
Sample Code													
Number of Samples		1	2	3	4	5	6	7	8	9	10	11	12
1 (OIL)	Amount of Additives % g/g	T1	T2	T3	T4	T5	T6	T10	T11	T12	T16	T17	T18
2		A1									XY3	XY3	XY3
3			A2		A2	A2	A2	A2	A2	A2	XY3	XY3	XY3
4				A3									
5					O1								
6						O2							
7							O12	O12	O12	O12	O12	O12	O12
8								K1					
9									K2		K2	K2	K2
10										K3			
11											F1		
12												F2	
13													F3
SRV Disc Aw	TESTS												
SRV EP													
F max													
4 Ball AW													
X RAY													
Rust Test													
RBOT													
Foam Test													

WP2: Design and Formulation Development

Testing and validation

- ✓ Test in accordance with technical specifications
- ✓ Trial sample transfer process
- ✓ Oil mist and OECD 301 tests
- ✓ Performing the tests of the formulation coded BRC022001-1, which is thought to lead to the final product, and determining that the results are within the determined target values,

It has been observed that the results obtained with the formulation in each metal type provide much better metalworking performance than TMP ester.

Test, Feature and Unit	Test Method	Target Value	BRC022001-1
Kinematic Viscosity(40°C, cSt)	ASTM D 445	10-500	56,52
Pour Point(°C)	ASTM D 97	max -10	-10
Flash Point(°C)	ASTM D 92	min 140	>140
Total Acid Number (TAN) (mg KOH/g)	ASTM D 974	max 5,0	2,0
FoamTest (mL/mL, St I)	ASTM D 892	max 50/0	30/0
SRV Wear Test (fmax) (for Al/Ti)	ASTM D 6425	max 0,500	0,195
SRV EP Step Test (N) (for Al/Ti)	ASTM D 5706	min 400	400
Tapping Torque Test for Al 6061 (Nm)	BLN 105	max 350	103,3
Tapping Torque Test for Al 7075 (Nm)	BLN 105	max 350	173,4
Tapping Torque Test Ti (Nm)	BLN 105	Max 350	142,6
Rust Test	ASTM D 665 A	Pass	Pass
Aluminum Staining Test	BLN 99	Pass	Pass
Wettability Test	BLN 172	Good	Good
Cleanability Test	BLN 147	Should be easy	Easy
Biodegradability Test	OECD 301	min %80	To be tested

WP2: Design and Formulation Development

Design of manufacturing process

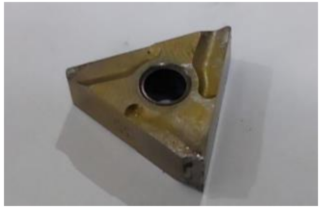
- ✓ During this assignment, the consortium created and discussed test plans to meet the project's objectives. These plans were determined by considering aluminum and titanium materials, cutting tools, manufacturing process and the MQL system used.

Titanium test specimens

WP2: Design and Formulation Development

Design of manufacturing process

- ✓ In this work plan, the consortium performed turning tests with different cutting tools to understand the cutting conditions, taking into account the variables/parameters (variations, uncertainties and independencies) in the manufacturing process.
- ✓ The results obtained were accepted as a starting point for the next stage tests.



Machining tap used in the preparation of part material.

Prop material in the first turning tests.

Prop material and machining tap

Probe material after preparation and test.

WP2: Design and Formulation Development

Design of manufacturing process

Project partners;

- ✓ Uses a 3D CMM equipment for dimensional verification, a microscope to check for homogeneity and changes in the surface of metallic parts and tools, and a rugosimeter to measure surface roughness.
- ✓ Analyzed the wear of the cutting tools used in the initial tests and defined procedures and criteria (edge wear) to measure this wear. In the next period, this wear will be measured under the microscope by paying attention to the lack of material on the instrument, the wear pattern will be analyzed and the nature of this wear will be interpreted.
- ✓ The wear that occurs in the first stage occurs in the form of polishing the tool or continuous wear of the material.

Microscope image used during measurement

Tool tap wear

WP2: Design and Formulation Development

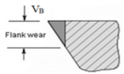
Design of manufacturing process

- ✓ The project partners created the parameter tables to guide the processing tests. It has also established procedures for checking and verifying the surfaces and dimensions of the tested parts.
- ✓ The consortium established procedures and specified specifications for monitoring and control of machined probes and verification of surfaces.

SMART EUREKA

Material to cut:	Ti6Al4V, AMS 4911 Ø40x60mm
Insert reference:	DCGX 55º / DCMT 55º
Insert material:	Hard metal
Insert coating:	None
Strategy:	Axial cut from Ø50 to Ø17.5mm
Machine/model:	Late Machine / Mori Seiki SL25

Radius insert:	0.2mm
ISO Grade insert:	N10/P10

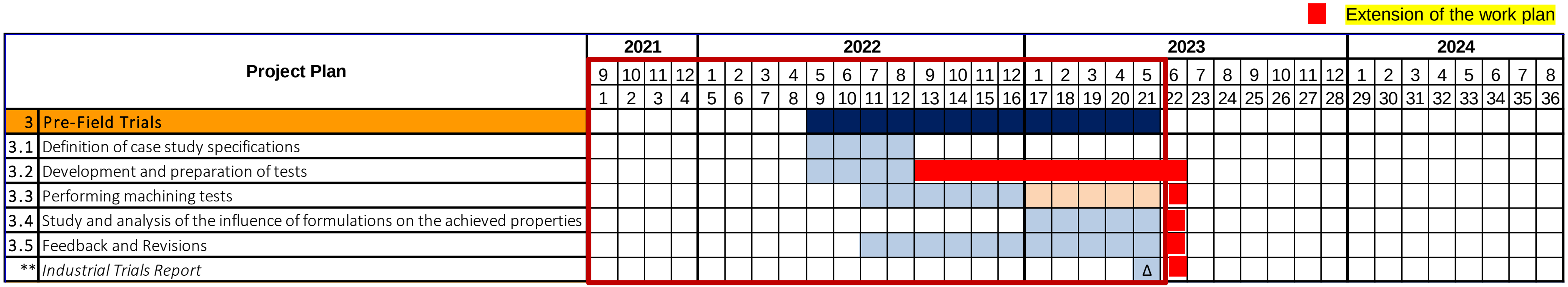


		Cutting Conditions					Results			
Tip tool n.	Experience n.	Tool Grade [Reference]	Cutting Speed [m/min]	Feed [mm/rev.]	Depth Cut [mm]	MQL / Emulsion [60/150]	Total Time [min]	Total Meters [m]	Ra Roughness [µm]	Flank Wear Vb [µm]
	1	N10 DCGX	60	0,1	0,3	Emulsion	25m 11s	1	Good	
	2	N10 DCGX	80	0,05	0,2	Emulsion	42m 27s	1,5	Good	
	3	N10 DCMT	80	0,1	0,05	Emulsion	54m 09s	6	Good	
	4	N10 DCMT	80	0,1	0,4	Emulsion	21m 00s	1,125	Bad	
	5	N10 DCMT	80	0,1	0,2	Emulsion	25m 29s	2,25	Medium	
	6	N10 DCGX	80	0,1	0,2	Emulsion	63m 25s	3,75	Good	
	7	N10 DCGX	80	0,1	0,4	Emulsion	9m 46s	0,9375	Medium	
	8									
	9									
	10									
Comments										

Table of Test Parameters

GANTT CHART

WP3: Pre-Field Trials



Task 3.1 : Definition of case study specifications (Task leader: Toolpresse, Task participants: Centimfe) M9-M12

Task 3.2: Development and preparation of tests (Task leader: Centimfe, Task participants: Toolpresse) M9-M12

Task 3.3: Performing machining tests (Task leader: Centimfe, Task participants: Toolpresse) M11-M21

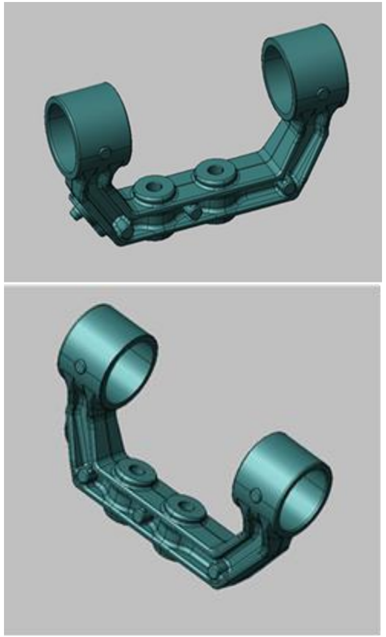
Task 3.4: Study and analysis of the influence of formulations on the achieved properties (Task leader: Centimfe, Task participants: Toolpresse and Belgin) M17-M21

Task 3.5: Feedback and Revisions (Task leader: Belgin) M11-M21

WP3: Pre-Field Trials

Definition of case study specifications

- ✓ The project partners (Toopresse and CENTIMFE) have prepared specifications for each geometric element of a 'Case Study Geometry' for the project objectives of developing a new lubricant.
- ✓ A CAD software was used to create the different elements of the complex geometry.
- ✓ Based on a specific complex geometry, this geometry will allow to demonstrate the feasibility of the proposed approach in an industrial setting at an advanced stage of the project.
- ✓ The definition of the case study configuration is to bring about a more sustainable metalworking process, taking into account the conditions and typology of the tests to be performed throughout the project and the purpose of validating the use of a new composition and new systems to provide cutting oil in the interface tool/part.



Case study geometry (Suggestion).

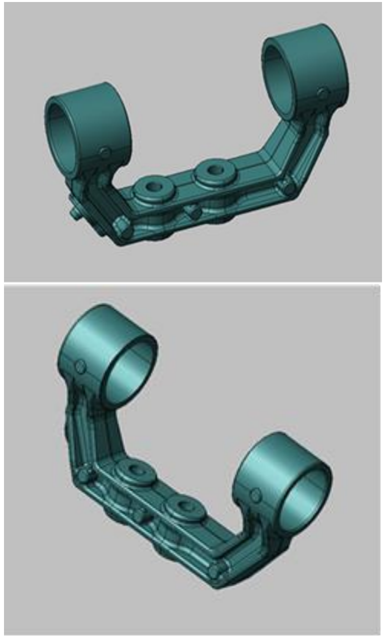
WP3: Pre-Field Trials

Definition of case study specifications

- ✓ In this business plan, geometry has been specially designed. A complex geometry has been designed to specify the most demanding machining parameters.

- ✓ The basic geometry is the geometry of a part available in industrial production by Toolpresse, one of Belgin's project partners, in the position of end-user. With this basic geometry, the consortium aims to provide real data generation to compare with results from tests with new formulations.

- ✓ In this work package, the consortium detailed, discussed and finalized the specifications for the development of the case study and all its components.



Case study geometry (Suggestion).

WP3: Pre-Field Trials

✓ **Task 3.2: Development and preparation of tests**

Work done	Upcoming Works
Preparation of DOE 1st phase of testing	Task 3.3 Performing machining tests
Design & production of an MQL system	
Proposal of an MQL system suitable for high viscosity fluids	Production & test of a new MQL system

WP3: Pre-Field Trials

✓ Task 3.2: Development and preparation of tests

Initial testing – Turning of Titanium

Material	Ti6Al4V, AMS 4911
Dimension, [mm]	Ø50 x 17,5
Tool Grade	Hard Metal, DCGX 55º
Cutting Speed, [m/min]	60; 80
Feed, [mm/rev.]	0,1
Depth Cut, [mm]	0,05; 0,2; 0,3; 0,4 (4 repetitions)
Emulsion	*Castrol HYSOL 39 CBF soluble oil at approximately 8% concentration

Main objectives	Understand the cutting process & Establish the starting parameters

WP3: Pre-Field Trials

✓ Task 3.2: Development and preparation of tests

DOE for Machining Tests with Titanium & Aluminium

	Turning Machining		Milling Machining	
	Titanium	Aluminium	Titanium	Aluminium
Oil O1	P1=1st phase	P2	P3	P4
Oil O2	P1=1st phase	P2	P3	P4
Oil O3	P1=1st phase	P2	P3	P4

WP3: Pre-Field Trials

✓ Task 3.2: Development and preparation of tests

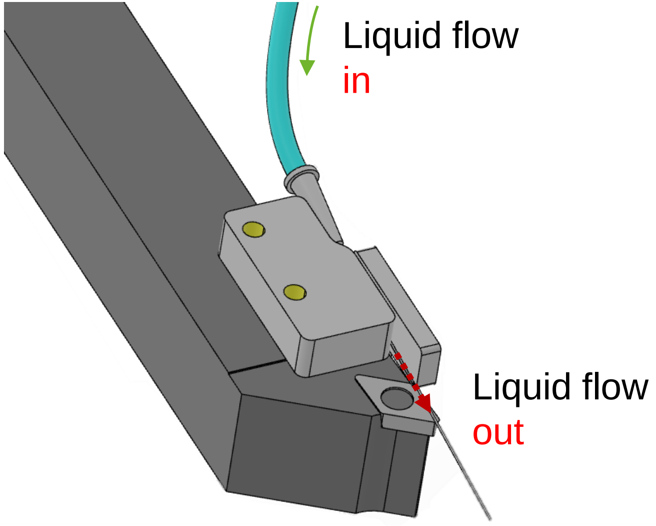
The machine tool to make the initial tests of Turning machining

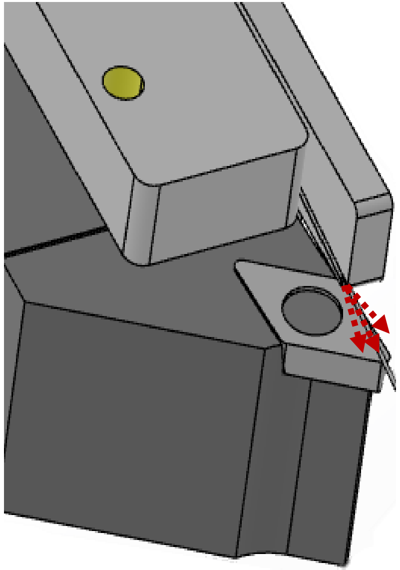
Turning Center MORI SEIKI SL25

WP3: Pre-Field Trials

✓ Task 3.2: Development and preparation of tests

Design and production of the new MQL system





Results:

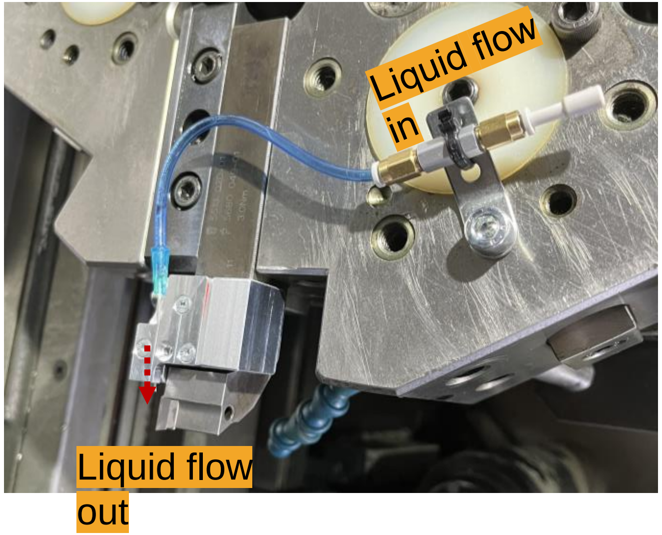
(Water or emulsion) Flow rate of 8 ml/min

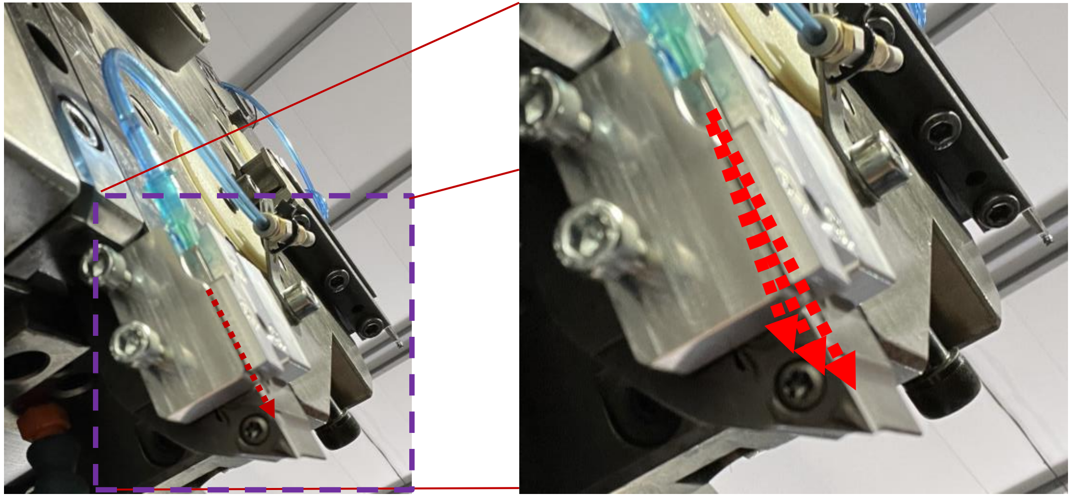
(New Oil) Flow rate ?Unknown? ml/min

WP3: Pre-Field Trials

✓ Task 3.2: Development and preparation of tests

The new MQL system





WP3: Pre-Field Trials

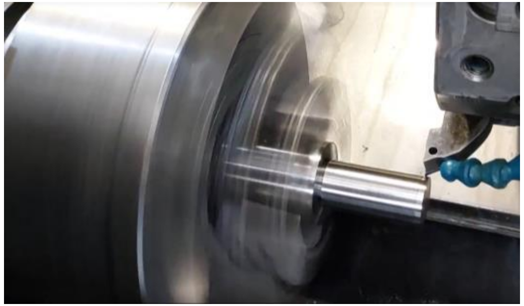
✓ Task 3.3 Performing machining tests

Work done	Upcoming Works
Initial tests	
1st phase of testing	2^{nd} phase of tests
	Discussion of results and proposals for improvement

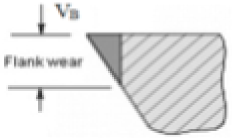
WP3: Pre-Field Trials

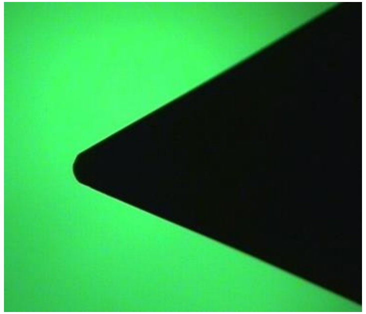
✓ Task 3.3 Performing machining tests

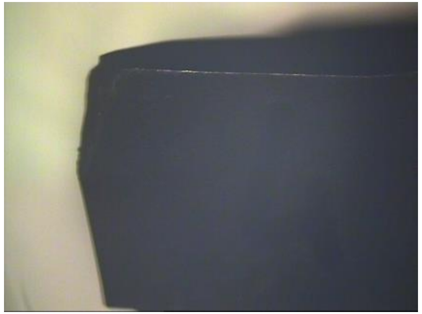
The initial tests with Titanium samples



Tool tip wear







WP3: Pre-Field Trials

✓ Task 3.3 Performing machining tests

1st phase of testing – Turning of Titanium

Results from the initial tests

Material	Ti6Al4V, AMS 4911
Dimension, [mm]	Ø50 x 34
Tool Grade	Hard Metal, DCGX 55º
Cutting Speed, [m/min]	80
Feed, [mm/rev.]	0,1
Depth Cut, [mm]	0,4; 0,8; 1,2; 1,6 (4 repetitions)
Emulsion	*Castrol HYSOL 39 CBF soluble oil at approximately 8% concentration

Main objectives	Understand the performance of the conventional oil
	Establish and refining the parameters to use in the different machining tests

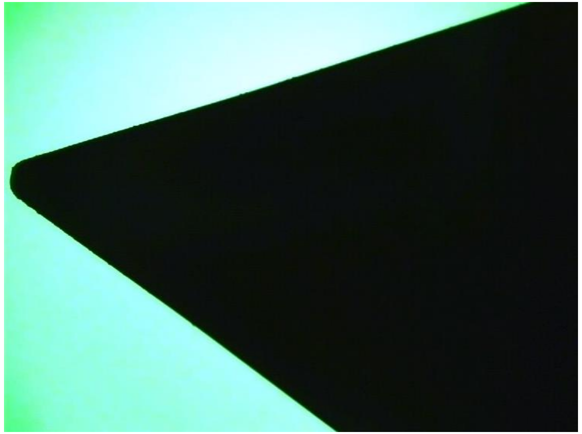
WP3: Pre-Field Trials

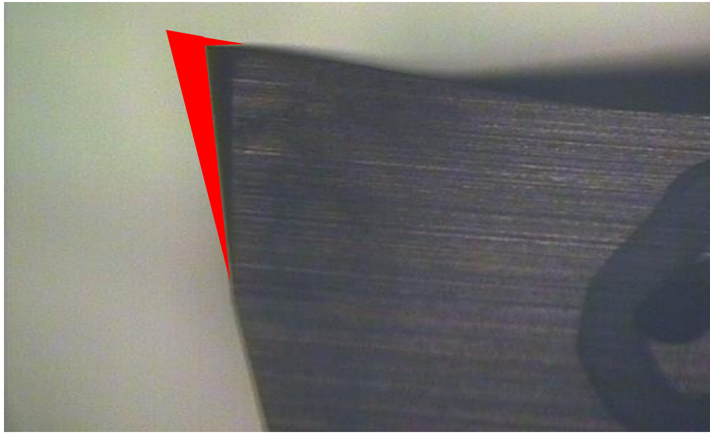
✓ Task 3.3 Performing machining tests

<u>1st phase of testing – Turning of Titanium</u>

Analysis of the tool wear

Tool tip wear



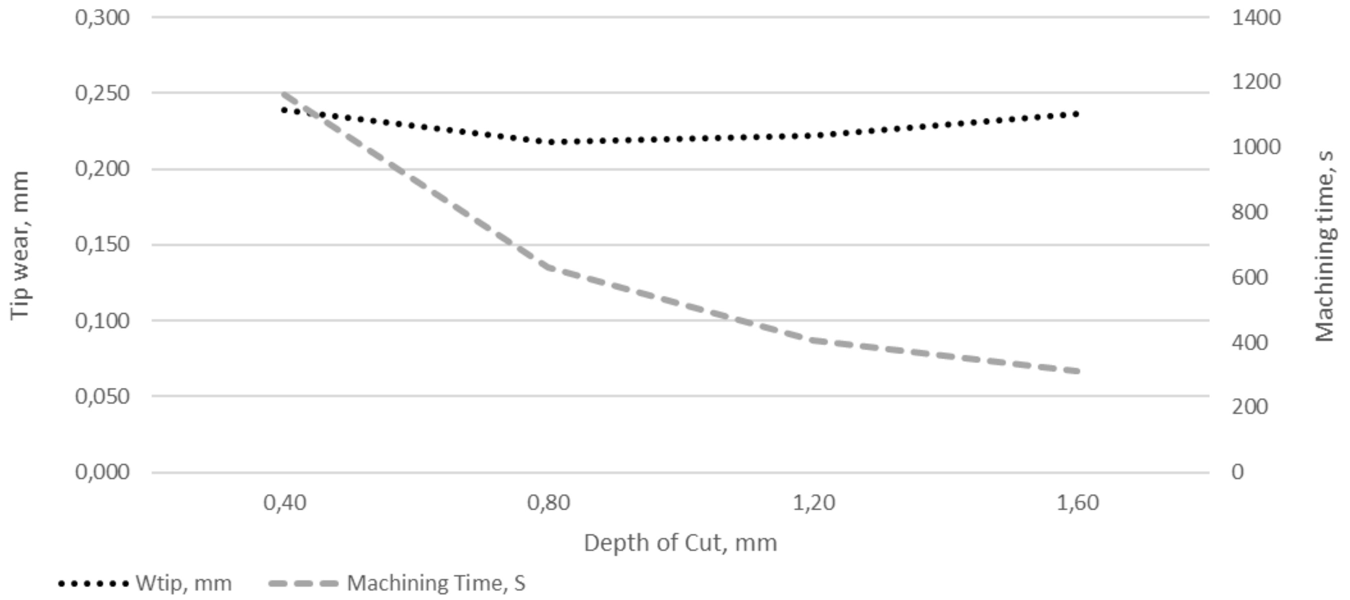


Tool tip

WP3: Pre-Field Trials

✓ Task 3.3 Performing machining tests

1st phase of testing – Turning of Titanium – Main conclusions



✓ Task 3.3 Performing machining tests

Phase 1 – Main conclusions

Roughness (Ra) remains in an average of around 0,83 microns for all samples

Wear of the tip remains between 0,23 to 0,25 mm for all samples

Cutting time is markedly reduced when depth of cut increases

At this stage, we conclude that we can work with the highest depth of cut value, as we gain time without compromising surface quality or tool wear.

WP3: Pre-Field Trials

✓ **Task 3.3 Performing machining tests**

<u>Phase 2</u>

Test the performance of the 3 oil samples received from BELGIN

Testing will begin on May 11

	Turning Machining		Milling Machining	
	Titanium	Aluminium	Titanium	Aluminium
Oil O1	P1	P2	P3	P4
Oil O2	P1	P2	P3	P4
Oil O3	P1	P2	P3	P4

WP3: Pre-Field Trials

Study and analysis of the influence of formulations on the achieved properties

- ✓ Collection of information from the tests carried out with the MQL system, taking account on cutting parameters on machining operations in the processing of aluminium and titanium alloys.

- ✓ Understand if the cutting fluid, in the proper amount and condition, can effectively lubricate and cool the tool/workpiece interface, and if this will increase tool life and surface quality.

- ✓ Feedback from the studies and working procedures will be created.

- ✓ Opinions, suggestions and contributions to optimize the new product.

WP3: Pre-Field Trials

Feedback and Revisions

- ✓ Studies with new generation polymeric esters in addition to TMP ester formulation studies
- ✓ Formulation studies and sample transfer process.
- ✓ It was determined that the product with the code BRC022001-2 exhibited the lowest and therefore the best metalworking performance on all metal alloys studied.

Test, Feature and Unit	Test Method	Target Value	BRC022001-2	BRC022001-3
Kinematic Viscosity (40°C, cSt)	ASTM D 445	10-500	222,42	208,3
Pour Point (°C)	ASTM D 97	max -10	<-10	<-10
Flash Point (°C)	ASTM D 92	min 140	>140	>140
Total Acid Number (TAN) (mg KOH/g)	ASTM D 974	max 5,0	<5	<5
Foam Test (mL/mL, Kd I)	ASTM D 892	max 50/0	30/0	20/0
SRV Wear Test (fmax) (for Al/Ti)	ASTM D 6425	max 0,500	0,147	0,192
SRV EP Step Test (N) (for Al/Ti)	ASTM D 5706	min 400	400	500
Tapping Torque Test Al 6061 (Nm)	BLN 105	max 350	82,8	91,6
Tapping Torque Test Al 7075 (Nm)			166,8	175,5
Tapping Torque Test Ti-6Al-4V (Nm)			119,6	126,0
Rust Test	ASTM D 665 A	Pass	Pass	Pass
Aluminum Staining Test	BLN 99	Pass	Pass	Pass
Wettability Test	BLN 172	Good	Good	Good
Cleanability Test	BLN 147	Should be easy	Easy	Easy
Biodegradability Test	OECD 301	min %80	To be tested	To be tested

WP4: Pilot Production and Validation

Project Plan		2021				2022															2023									2024							
		9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
4	Pilot Production and Validation																																				
4.1	Small Batch Production																																				
4.2	Testing and validation																																				
4.3	Cost Evaluation																																				
4.4	Environmental Impact Analysis																																				
**	*Optimum process conditions*																						Δ1														Δ2

Task 4.1: Small Batch Production (Task leader: Belgin) M20 – M24

Task 4.2: Testing and validation (Task leader: Belgin) M22-M30

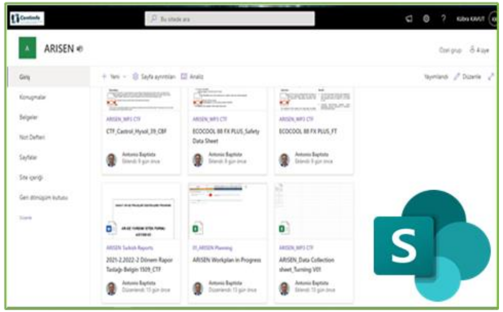
Task 4.3: Cost Evaluation (Task leader: Belgin, Task participants: Centimfe and Toolpresse) M17-M36

Task 4.4: Environmental Impact Analysis (Task leader: Belgin, Task participants: Centimfe and Toolpresse) M17-M36

WP5: Project Management

Project Plan		2021				2022												2023												2024							
		9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
5	Project Management																																				
5.1	Business Division, Management Plan and Other Sharing Between Partners																																				

- ✓ Project consortium agreement.
- ✓ Project management group.
- ✓ Project Monthly meetings.
- ✓ Microsoft SharePoint



MEETING MINUTES

BELGiN

MEETING	MONTHLY MEETING of ARISEN
MEETING DATE	04.08.2022
MEETING TIME	16:00-17:00 (TRT)
MEETING PARTICIPANTS	Kerem BELGİN, Hanife GÜLEN TOM, Kübra KAVUT, Martinha SOARES, Rui SOARES, Antonio BAPTISTA, Jose FILIPE

Agenda: PCA, Reporting Processes, Gantt Chart Term Studies

No	Topic	Action	Responsible	Deadline
1	PCA	The signing process of the PCA document was completed by Centimfe and Toolpresse. As soon as the documents reach Belgin, the signature process will be completed and 2 copies will be sent to the Portuguese partners.	BELGIN	12.08.2022
	Reporting Processes	TUBITAK REPORT; Based on the submission of the report of the 6-month studies. The 1st between SEPTEMBER & DECEMBER 2021 The 2nd report between JANUARY & JUNE 2022. Belgin shared report drafts with partners via Microsoft SharePoint. The partners created the information requested from them on the report. They will also add the work they have done for the missing 2.6, 3.1 and 3.2 to the reports. For Task 3.3, Belgin will deliver samples to partners for testing by mid-September.	BELGIN	30.09.2022
		ANI REPORT; According to ANI, the Portuguese partnership must send: The 1st report between SEPTEMBER 2021 & AUGUST 2022 If Portuguese partners share their report drafts via Microsoft SharePoint, Belgin will create the requested information on the report.	CENTIMFE TOOLPRESSE	30.09.2022
		SMART REPORT; Partners will start working to create the Smart report. The draft of the presentation was shared via Microsoft SharePoint. All three partners will quickly complete the writing of the report.	BELGIN CENTIMFE TOOLPRESSE	31.08.2022

WP6: Dissemination and exploitation activities

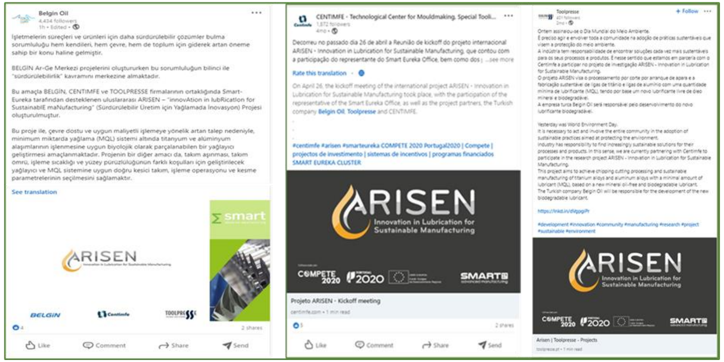
Project Plan		2021				2022												2023												2024							
		9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8	9	10	11	12	1	2	3	4	5	6	7	8
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
6	Dissemination and exploitation activities																																				
6.1	Dissemination and exploitation activities																																				

- ✓ Logo design.
- ✓ Logo selection
- ✓ This logo is used throughout the project to represent the image of the project in different fields and documents.

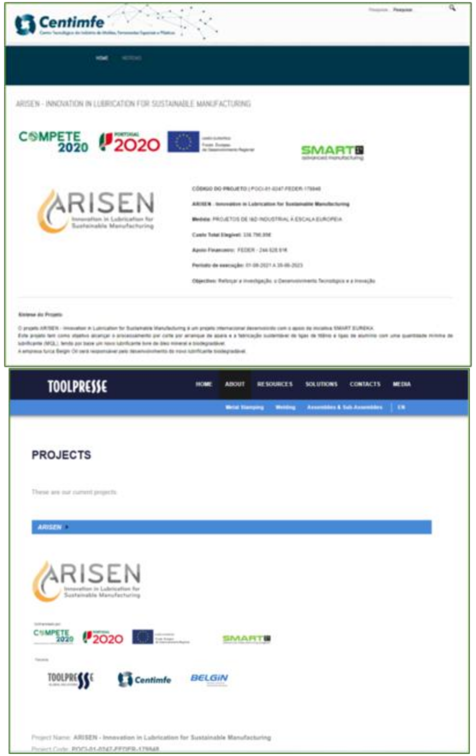
ARISEN Logo Examples.

WP6: Dissemination and exploitation activities

- ✓ Sharing a tab of the project on the partner's websites.
- ✓ Sharing posts on the partner's LinkedIn accounts to announce and promote the project and its activities.
- ✓ The process of creating a web page and LinkedIn account for the project continues.



Consortium LinkedIn posts.



Project web tab view of Centimfe and ToolPresse.

WP6: Dissemination and exploitation activities

✓ 1 technical article and 2 articles were published in the International Refereed Journal, and 2 international congress and symposium activities were attended.

1) Publication of a technical article:

- Article Title: Contribution to the sustainable production of metal parts
- Magazine Name: Magazine O MOLDE Nº135,
- Volume: Ekim 2022
- Authors: António Baptista[1], Martinho Soares[2], Kübra Kavut[3], Hanife Gülen Tom[3]
- Author Definitions: 1:CENTİMFE, 2:TOOLPRESS, 3: BELGİN


UM CONTRIBUTO PARA A PRODUÇÃO SUSTENTÁVEL DE PEÇAS METÁLICAS

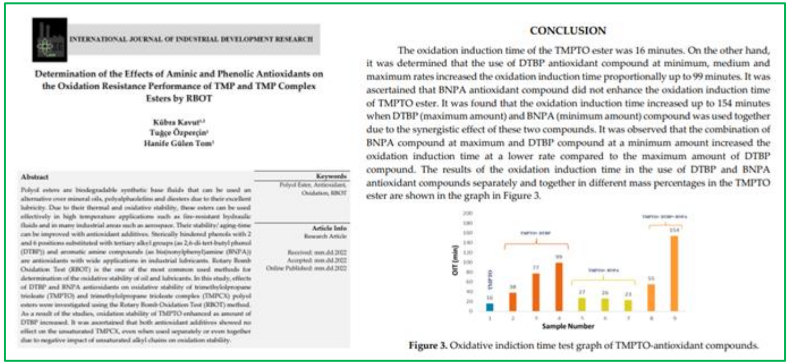
ARISEN

WP6: Dissemination and exploitation activities

2) Publication of an article in the International Refereed Journal:

- Article Title: Determination of the Effects of Aminic and Phenolic Antioxidants on the Oxidation Resistance Performance of TMP and TMP Complex Esters by RBOT
- Journal Name: International Journal of Industrial Development Research (IJIDR)
- Volume: December 2022
- Authors Kübra Kavut[1], Tuğçe Özperçin[1], Hanife Gülen Tom[1]
  Author Definitions: 1: BELGİN

- Article Title: Comparison of the Effects of Vegetable Oils such as Castor and Canola Oil and Polyol Esters on Metalworking Performance of AL 6061 and Al 7075 Metal Alloys by Tapping Torque Method
- Journal Name: The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2022
- Volume: Vol. 21, Pages 248-257.
- Authors Kübra Kavut[1], Tuğçe Özperçin[1], Hanife Gülen Tom[1]
- Author Definitions: 1: BELGİN



INTERNATIONAL JOURNAL OF INDUSTRIAL DEVELOPMENT RESEARCH

Determination of the Effects of Aminic and Phenolic Antioxidants on the Oxidation Resistance Performance of TMP and TMP Complex Esters by RBOT

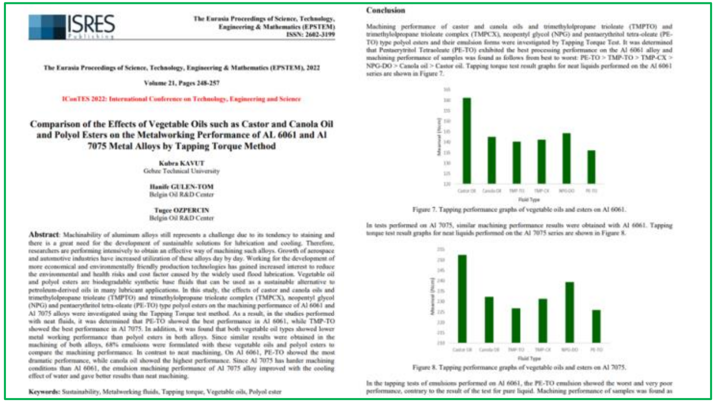
Kübra Kavut
Tuğçe Özperçin
Hanife Gülen Tom

CONCLUSION

The oxidation induction time of the TMPTO ester was 16 minutes. On the other hand, it was determined that the use of DTBP antioxidant compound at minimum, medium and maximum rates increased the oxidation induction time proportionally up to 99 minutes. It was ascertained that BNPA antioxidant compound did not enhance the oxidation induction time of TMPTO ester. It was found that the oxidation induction time increased up to 154 minutes when DTBP (maximum amount) and BNPA (minimum amount) compound was used together due to the synergistic effect of these two compounds. It was observed that the combination of BNPA compound at maximum and DTBP compound at a minimum amount increased the oxidation induction time at a lower rate compared to the maximum amount of DTBP compound. The results of the oxidation induction time in the use of DTBP and BNPA antioxidant compounds separately and together in different mass percentages in the TMPTO ester are shown in the graph in Figure 3.

Figure 3. Oxidative indiction time test graph of TMPTO-antioxidant compounds.

IJIDR Article image.



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The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM)

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2022

Volume 21, Pages 248-257

IConTES 2022: International Conference on Technology, Engineering and Science

Comparison of the Effects of Vegetable Oils such as Castor and Canola Oil and Polyol Esters on the Metalworking Performance of AL 6061 and Al 7075 Metal Alloys by Tapping Torque Method

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Conclusion

Machining performance of castor and canola oils and trimethylolpropane trioleate (TMPTO) and trimethylolpropane trioleate complex (TMPCX), neopentyl glycol (NPG) and pentaerythritol tetra-oleate (PE-TO) type polyol esters and their emulsion forms were investigated by Tapping Torque Test. It was determined that Pentaerytritol Tetraoleate (PE-TO) exhibited the best processing performance on the Al 6061 alloy and machining performance of samples was found as follows from best to worst: PE-TO > TMP-TO > TMP-CX > NPG-DO > Canola oil > Castor oil. Tapping torque test result graphs for neat liquids performed on the Al 6061 series are shown in Figure 7.

Figure 7. Tapping performance graphs of vegetable oils and esters on Al 6061.

Figure 8. Tapping performance graphs of vegetable oils and esters on Al 7075.

EPSTEM Article image.

WP6: Dissemination and exploitation activities

3) National/ International Symposium-Congress-Conference Participation and Paper Presentations:

- Paper Title: Comparison of the Effects of Vegetable Oils such as Castor and Canola Oil and Polyol Esters on the Metalworking Performance of AL 6061 and Al 7075 Metal Alloys by Tapping Torque Method-
- Congress/Conference/Symposium Name: 6th International Conference on Technology, Engineering and Science (IConTES)- Date: 16-19 November 2022
- Authors: Kübra Kavut[1], Tuğçe Özperçin[1], Hanife Gülen Tom[1] Author Definitions: 1: BELGİN

- Paper Title: Determination of the Effect of TPPT on the Anti-wear Performance of Vegetable Oils and Polyol Esters
- Congress/Conference/ Symposium Name: EGE 8th INTERNATIONAL CONFERENCE ONAPPLIED SCIENCES
- Date: 25 December 2022
- Authors: Tuğçe Özperçin1, Kübra Kavut1, Hanife Gülen Tom1
- Author Definitions: 1: BELGİN

IConTES presentation image.

EGE 7TH INTERNATIONAL CONFERENCE ON APPLIED SCIENCES - DECEMBER 24-25,2022 - IZMIR

DETERMINATION OF THE EFFECTS OF TPPT ON THE ANTI-WEAR PERFORMANCE OF VEGETABLE OIL AND POLYOL ESTERS

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ABSTRACT

Design of green process is the key points of industries for sustainable energy and environmental protection. Replacement of petroleum derived lubricants, which are extensively used to reduce wear and friction in the mechanical systems, with bio-lubricants is one of the crucial steps for achievement of this criteria. Vegetable oils and polyol esters are promising feedstocks for bio-lubricant formulations. Although vegetable oils are abundant and they have low cost, they have limited utilization since their low temperature behavior and thermo-oxidative stability are poor. On the other hand, polyol esters have high thermal and oxidative stability. These base fluids can be formulated with extreme pressure (EP) and anti-wear (AW) additives in metal working application for better machining performance. Triphenyl phosphorothionate (TPPT) is one of the promising environmentally friendly anti-wear additives that is used in biodegradable lubricant formulations. Canola vegetable oil is commonly used in industrial application due to its high annual production. Trimethylolpropane trioleate (TMPTO) and pentaerythritol tetra-oleate (PE-TO) polyol esters have wide acceptance as lubricant base fluid due to their excellent lubricity and stability. Based on these informations, this study presents an investigation of effect of TPPT on tribological performance of canola oil, TMPTO and PE-TO by using four ball method. For this purpose, wear-scar diameters of pure form of vegetable oil and polyol esters and their formulations with two different amount TPPT compared. As a result, it was determined that low TPPT amount (0.5%) has no effect on anti-wear performance whereas high TPPT amount (2 %) enhanced the tribological performance of formulations. Lowest wear scar diameter was obtained from the formulations of TMPTO with 2 % TPPT.

Keywords: vegetable oil, polyol ester, anti-wear additives

Paper image.

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