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# About the Use of mineral and vegetable Oils to improve the Sustainability of Steel Quenching

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#### Abstract

The Die Assisted Oil Quenching process is a highly customisable heat treatment that permits a relevant reduction of distortions thus limiting or avoiding following grinding operations. Distortion reductions can be obtained not only by an action on process parameters and equipment but also by choosing suitable quenching media.

Nowadays, the most widely used quenching fluids are mineral oils. Nevertheless, a number of innovative quenchants that are derived from oily plants are available on the market as possible alternatives to such media. Such multiplicity leads to a decision-making problem that may deeply affect the global sustainability of the process.

In this paper, a comparative study between a mineral and a vegetable oil is performed. A preliminary analysis of the environmental impacts of the two quenching media is presented. Then, an experimental activity has been performed to investigate the technical performances of the two media in terms of dimensional and metallurgical properties of the quenched parts. Furthermore, a measurement of oily fogs has been performed during quenching to verify possible emission reductions.

The experimental activity pointed out a better control of part distortions and a comparable metallurgical microstructure when vegetable oil is used for quenching. In addition, no oily fogs have been observed for this quenching medium.

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Keywords: Die Assisted Oil Quenching; Mineral Oil; Vegetable Oil; Distortion;

## 1. Introduction

The constant world increase in population and wealth contributes to determining a continuous rise in the demand for goods and thus in the consumption of natural resources. The global market and the present need for a growth of manufacturing activities, as a consequence, determined the need for designing more sustainable patterns for humankind development. In this direction, the development of processes with a high know-how content can be helpful if the sustainability issue is considered and satisfied [1] [2].

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The sustainable manufacturing paradigm, in particular, aims to the fulfilment of the three pillars of the economy, society and environment to achieve the sustainability of industrial processes [3]. Recently, the definition of sustainable manufacturing has been theorised through the "6R" concept that leads directly to the circular economy in which resources undergo a closed loop [4]. This paradigm stresses the need to re-define present products or services to get the same quality in a cheaper way and through the use of a smaller amount of raw (and possibly renewable) materials [5]. On the other hand, societal aspects regarding the wide-spreading of human rights are enhancing the standards of life; whereas, environmental attributes deal with the need to make use of materials for more than one time according to the 6R configuration [3]. This discussion drives to understanding that the sustainable pillars are unequivocally connected amongst them and the achievement of one promotes the obtainment of the others.

In this scenario, the main objective of production engineering must be to find out solutions to reduce the environmental impact of manufacturing processes and enhancing the condition of manpower without affecting the quality of the final product. A particular attention has to be paid to those productive steps presenting high energy consumption and polluting emissions; an example is heat treatment of steel, which is a fundamental phase in the production cycle of many mechanical parts.

Along the technological chain of steel mechanical parts, quenching operation has been revealed as a strategical production step to obtain mechanical performances by modifying the microstructure of materials. To accomplish the sustainable manufacturing issue in heat treatments means not only the optimisation of the heating phases but also the adoption of adequate equipment and the selection of the most suitable cooling fluids (possibly through the use of renewable resources [6]). One of the most important technical issues of quenching processes comes from induced part distortions. The main reasons for this phenomenon are non-uniform thermal gradients, which are coming across the cooling phase, and volume variations that are associated with material phase transformations [7] [8].

The Open Tank Oil Quenching (OTOQ) heat treatment handles a single batch of products where inevitably unequal cooling conditions are present and dimensional modifications cannot be avoided. Then, the design of quenched parts has to consider machining allowances in order to maintain the components within dimensional tolerances by machining operations (typically grinding) with an increase in times and costs of the production cycle. The adoption of a more controllable and replicable quenching treatment is a way to improve the process sustainability because not only the reduction of machining allowances but also the avoidance of following manufacturing operations can be achieved.

The Die Assisted Oil Quenching (DAOQ) technique permits the hardening of axisymmetric parts and aims at the reductions of dimensional and shape variations by means of a hydraulic press and a die. In Fig. 1 (a) is shown a typical scheme of the mentioned process. It consists of a quenching stage where a part is at the same time mechanically constrained through a pressure that is exerted by the mould and rapidly cooled down by means of a quenching medium.

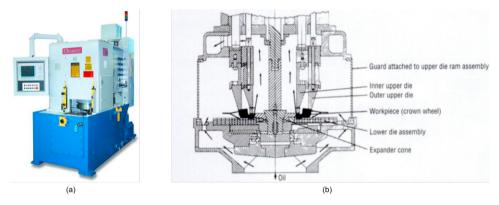


Fig. 1. (a) Hydraulic press; (b) Quenching mould for the Die Assisted Oil Quenching (DAOQ) technique [9]

The mould permits the control of critical component dimensions through an expander cone, which limits the inner hole of the treated part, and both an inner and an outer upper die, which act on the outer diameters thus allowing the handling of the planarity tolerances, see Fig. 1(b). The equipment ensures the standardization of the cooling phase by reducing thermal gradients and thus distortions. As an example, this process is particularly well-effective when

applied to gears and bearing rings.

Even if the cost of DAOQ is usually higher in comparison with OTOQ [9], benefits coming from a more controlled quenching process lead to drastically reduce machining allowances so that grinding operations can be significantly reduced or even completely avoided. As a result, cost, manufacturing time and wasting of material can be reduced. In the present paper, an investigation into the performances of two quenching media applied to DAOQ is presented. In particular, two quenchants derived from mineral and vegetable resources have been compared. As a first step, the environmental impact of the two media is discussed to identify the benefits and drawbacks of both the solutions. Then, an experimental activity will be presented to verify the effectiveness of the vegetable oil in obtaining required microstructures while limiting distortions. A measurement of oily fogs during the process is reported to show the advantages coming from the use of innovative quenchant in reducing polluting emissions.

## 2. Vegetable Oils for Quenching Purpose

The quenching process of steel components determines the achievement of the martensite microstructure, which is necessary to obtain high mechanical performances. In the meanwhile, unavoidable changes of the part shape and dimensions occur [10]. Quick heat extractions and low-temperature gradients are fundamental aspects in order to obtain a homogeneous martensite transformation and thus a reduced level of those distortions [7]. For an effective control on microstructure transformations, the cooling conditions must assure the highest cooling rate above  $M_s$  and the lowest one between  $M_s$  and the martensite finish temperature  $M_f$  [8]. These conditions strongly depend on adopted quenchants.

Quenching processes can be carried out by means of a wide assortment of cooling agents. The choice depends on a number of factors and, among them, processed materials, part geometries and design requirements. Traditionally, quenching media derive from the petroleum industry and the base-stocks are usually characterized by either naphthalene or paraffin that are added with additives to obtain appropriate performances. The oil should perform by avoiding the vapour formation because, otherwise, this phenomenon reduces dramatically the heat exchange coefficient at higher temperatures; then, the fluid agitation becomes an important parameter to be controlled to remove the vapour phase. Viscosity and wettability are two other important quenchant characteristics. As a result, a trade-off has to be identified to match conflicting process requirements and to obtain high effective quenchants.

A sustainable approach to manufacturing has to include not only technical aspects but the whole cycle of life of products or means of production. In this direction, nowadays, innovative quenching oils have been developed because a possible reduction of impacts can be achieved by the use of vegetable oils that are prepared from renewable sources [11] [12] [13].

With the mentioned purpose a preliminary comparison has been done to understand the main characteristics and differences between innovative quenchant formulations based on oil extraction from vegetables and mineral oils.

Two quenchants have been compared; the first one is a petroleum-based formulation (named "tsp6") and the second an innovative one (named "tempro G Bio Plus").

## 2.1. Cooling curve analysis

A technical comparison can be done based on the so-called cooling test for evaluating the cooling rate as a function of the temperature. Fig. 2 shows the experimental curve for both the oil formulation [12] [14]. It is worth to mention that the "tempro G Bio Plus" displays a reduced vapour formation at a higher temperature and the maximum cooling rate value at about  $640^{\circ}C$  instead of at about  $575^{\circ}C$  for the mineral oil. Both this aspects are relevant in order to achieve an optimised performance of the quenchant.

## 2.2. Impact comparison

In [15] a Life Cycle Assessment has been carried out by comparing petroleum-based diesel oil with soy-based lubricant. Only the production chain has been considered without additives. Agriculture, crushing and refining have been taken into account for the production of vegetable medium whereas extraction, finish and refining have been accounted for the mineral based oil.

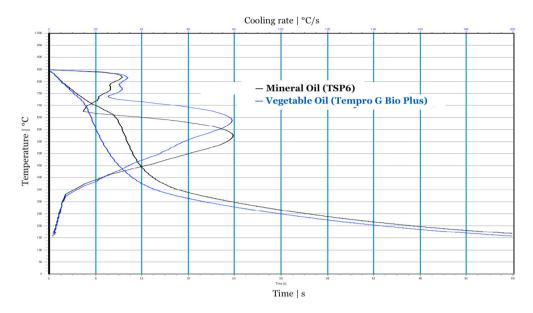


Fig. 2. Cooling rate as a function of the oil temperature. Experimental curves of the vegetable (blue curve) and mineral (black curve) quenching medium

The soy-based oil presented lower impact evaluations in terms of global warming, acidification potential, water intact, air pollution and human toxicity. On the other side, the eutrophication potential is higher for the case of vegetable quenching media; this could be also related to the use of unsafe substances such as pesticides that modify soil conditions. The impact assessments of vegetable quenching media are also correlated to the working method implied for the growing of the oily based plants as well as to the in-working conditions that can change the oil properties. So those innovative fluids have to be appropriately modified with additives especially for increased oxidation resistance [12], [16].

The main sustainability indicators have been modelled by means of a LCA regarding the production of the vegetable quenching oil. Agriculture and crushing have been determined by utilising bibliography data [17] and by considering the production of the investigated oil equals to the data presented in the reference study for the soybean quenchant [17]. Refining stage has been set taking into account real data obtained by the oil manufacturer without esteeming the additive influences. The LCA of the mineral quenching oil has been also deducted from [17]. Fig. 3 shows the adopted structure of the LCA that was implemented by the Open LCA software. Fig. 4 displays main results about the impact comparison between vegetable and mineral media.

It is worth to underline that the climate change (GWP100) records a negative sign for the vegetable quenchant, which means a life cycle for the oily plant that permits the absorption of carbon dioxide and a cut down of the GWP100 indicator. The mineral oil also shows a low GWP100 impacts that means the high standards utilised for the production of the mineral quenching oil [17].

Ozone Layer Depletion (OLD) and Acidification potential could be reduced by means of the vegetable quenching oil as reported in Fig. 4.

## 3. Experimental Activities

Sustainable manufacturing promotes improvements of processes and products in order to achieve a better working environment by enhancing the manpower value without compromising manufacturing results. The following experimental activity investigates the performances of mineral and vegetable oil for quenching pointing out mechanical and metallurgical outcomes, geometrical results and assessment of quenching fumes.

Conical bevel gears made of steel have been considered as the sample geometries. Three geometries have been con-

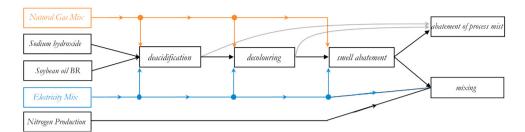


Fig. 3. Open LCA software model for a soybean vegetable oil

Results from LCA	Vegetable oil	Mineral oil
GWP100 Climate Change (kg CO 2 eq.)	-1.34	0.97
OLP Ozone layer depletion (kg CFC-11 eq.)	7.30E-09	7.6E-07
Acidification potential-average Europe (kg SO 2 eq.)	0.01	0.03

Fig. 4. Comparison between mineral and vegetable oils for relevant impacts

sidered and characterised by means of the following parameters: the inner diameter  $(d_i)$ , the ratio between  $d_i$  and the outer diameter  $(d_e)$  as well as the proportion between the part thickness (s) and  $d_i$ , see Fig. 5(a).

	di (mm)	Ig01 di/de	Ig02 s/di	Carburising Steels	
Geometry1	159,60	0,59	0,23	20MnCr5	
Geometry2	159,60	0,53	0,23	19MnCr5	
Geometry3	116,00	0,48	0,32	19MnCr5	
		(a)			(b)

Fig. 5. Identification of the investigated conical bevel gears (a); measurement equipment for the evaluation of the planarity tolerances (b)

Three batches - one batch for each considered geometry - composed of a total amount of 420 mechanical components - 140 part/batch - have been submitted to the carburizing phase ( $Ph_{01}$ ), then to the DAOQ process ( $Ph_{02}$ ) and, finally, to temper ( $Ph_{03}$ ). Each half batch was hardened by the use of mineral oil (210 parts, 70x3 specimens) and the rest by the vegetable-based medium. An in-line process control has been realized by means of dimensional analyses of each component after  $Ph_{02}$ . The dimensional analyses were carried out by means of manual micrometres and altimeters for the evaluation of diameters, thicknesses and planarity tolerances, as showed in Fig. 5(b).

A specific standard for the measurements has been established by defining characteristic detection points. 5+5 parts per batch out of the 70+70 components per batch have been randomly selected to be submitted to a further dimensional control after the carburizing phase (before the DAOQ process) and after the tempering phase (after the DAOQ process). In the following sections, this particular selection of parts has been named the "TOP 5".

Metallurgical samples were analysed through an optical microscope in order to determine the final material microstructure.

Quenching fumes have also been detected by means of a probe through a by-pass installed above the quenching chamber that captured directly the fumes from the hardening zone. Oily fog and CO contents have been measured in-process.

# 4. Results and discussion

In order to estimate dimensional changes, various inspections and measurements have been carried out during the production cycle of conical bevel gears by generating bulk evolutions for subsequent manufacturing operations. The TOP5 selection (see par. 3) permitted statistical representations of the collected data through mean values and standard deviations. As a first evaluation, a t-test has been done in order to verify that the two groups of dimensional measurements, which have been obtained trough mineral or vegetable quenchants, are independent (see par. 4.1).

## 4.1. T-test evaluation

The mentioned t-test has been carried out for the TOP5 selection by comparing the dimensions of each controlled geometrical characteristics after  $Ph_{01}$ ,  $Ph_{02}$ ,  $Ph_{03}$ .

The t-critical value has been set for the two sides t-distribution with 0.95 as the probability reference [18]. The comparison has generated eight degrees of freedom (type ) for the experiment thus resulting in a critical value equal to 2.3.

Table 1 collates, for each geometrical investigation, the corresponding t-test parameter that has to be matched with the threshold value. These variations point out the impact of the quenching oils on to the detected geometrical characteristics ( $Ph_{02}$ ).  $Ph_{02}$  analysis is also reported for a comparison.

TOP5	Geometry 1	Geometry 1	Geometry 2	Geometry 2	Geometry 3	Geometry 3
selection	t-value	t-value	t-value	t-value	t-value	t-value
Detected phase	$Ph_{01}$	$Ph_{02}$	$Ph_{01}$	$Ph_{02}$	$Ph_{01}$	$Ph_{02}$
inner diameter	0.1	3.3	-1.2	-1.7	0.0	-1.8
outer diameter	-1.3	2.2	0.5	3.3	-0.1	3.3
thicknesses	1.9	0.0	-2.6	1.9	0.9	1.8
planarity tolerance	-0.3	3.6	-1.1	1.0	2.1	0.6

Table 1. Test t Analysis

The outer diameters was practically always affected by the use of mineral or vegetable quenching oil. The inner diameter of geometry 1 has the same behaviour while geometry 2 and geometry 3 shows a different result. Thicknesses do not show relevant variations (in comparison with the  $Ph_{01}$ ). Planarity tolerances present a relevant variation only in case of the geometry 1.

The DAOQ equipment controls and modifies the heat extraction by means of a direct metallic contact between the part and the die surfaces, which are cooled through the quenchant. The process outcome is then connected not only to the quenching media but also to the process equipment and related parameters (die contact-surface and pressure, oil flux and pressure, etc.). In this case, the DAOQ process allowed, particularly, the control of some distortion magnitudes (or the planarity and thicknesses) while the effect of different cooling oils is evident on the dimension that are not influenced by the constrained heat flux (or the inner and outer diameters).

# 4.2. Metallurgical inspection and hardness level

The cooling curves (Fig. 2) of investigated oils show comparable trends also if relevant differences are present. It implied that, concerning the cooling severities of the considered media, no large dissimilarities have been observed. In fact, both the oils determined the formation of the martensite microstructure after the DAOQ process. Furthermore, the case hardening depths shows overlapped trends thus implying the same mechanical results in terms of superficial hardnesses (equal to 60 HRC) and process effectiveness due to the absence of sharp hardness variations, see Fig. 6(b). Comparable hardening depths point out the effectiveness of the vegetable quenching oil on the heat treatment outcomes.

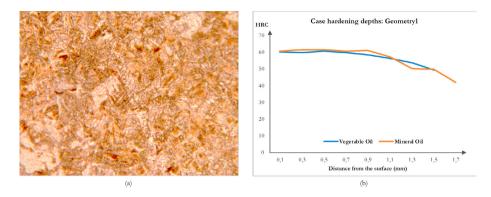


Fig. 6. Martensite microstructure obtained by the usage of mineral and vegetable quenching oils; (b) Experimental evaluation of superficial hardnesses and case depths for both mediums

#### 4.3. Analysis of part dimensions

After the carburising phase, due to the absence of process dissimilarities at his stage of the manufacturing cycle, no relevant dimensional variations were reported for the considered geometrical characteristics thus leading to the same starting conditions for the quenching phase. After the DAOQ process, it is worth to notice that the inner hole of geometry 1 was strongly increased. By starting from equal conditions after the carburising phase, all the dimensions unavoidably increased. In case of the vegetable quenching oil, the inner diameter variation, for example, was lower than the one determined by the mineral medium, Fig. 7(a) but, most important, a more accurate quenching stage as the in-process analysis clearly evidences in Fig. 7(b).

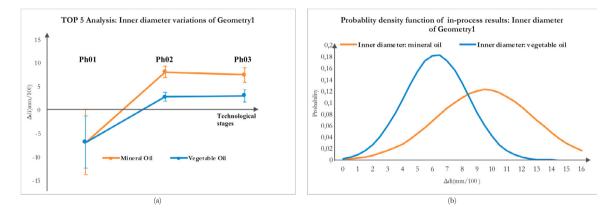


Fig. 7. (a) TOP 5 analysis: Evolution of inner diameters of geometry1 along the process stages; (b) In-line process control: Gaussian distributions of distortions regarding the inner diameters of geometry1

Summarizing, the renewable medium achieves an equal metallurgical microstructure, Fig. 6, and equivalent mechanical results by increasing the possibility of dimension control, Fig. 7.

## 4.4. Oily fog measurements

Samplings of quenchant fumes have been realized for one hour through an in-process experimental measurement for both the considered oils. The vegetable quenching medium put in evidence the presence CO (1.7 mg/Nm<sup>3</sup>) and the absence of oily fogs (less than 0.01 mg/Nm<sup>3</sup>). The mineral oil shows both the pollutants into the emission and, particularly 1.8 mg/Nm<sup>3</sup> of CO and about 1.2 mg/Nm<sup>3</sup>. By considering a yearly scenario (220 day/year and 8 h/day), the mineral oil produces 7 kg/year of oily fog. This aspect is relevant in order to reduce the need of air purification before its re-introduction in the environment.

## 5. Conclusion

Through a direct comparison, the paper investigates the use of a conventional mineral-based quenchant and an innovative vegetable-based one for heat treatment purpose of mechanical parts. The same mechanical and metallurgical results have been achieved but, above all, a better control of specific crucial dimensions, like for example the inner diameter of the considered conical gear, have been obtained for the vegetable oil. A preliminary LCA analysis put in evidence some possible points of strength for this production media. Besides, the environmental impacts have been analysed by taking into account a typical production cycle and through the measurement of emitted pollutants, like oily fogs and carbon monoxide, that must be controlled and purified before the reintroduction of the air into the environment.

The presented work suggested that innovative vegetable-based oils could be a good choice and a starting point to turn the heat treatment production plant into a more sustainable working environment.

A more detailed LCA is necessary in order to completely understand the possible advantages and weaknesses of both the solution. Further tests and investigations are necessary in order to discover the real potential of these quenchants.

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