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# ROADMAP SECTOR 3 IMPLEMENTATION OF MICROSYSTEMS IN THE WINE & BEER SECTOR

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The research leading to this publication has received funding from the European Union's Seventh Framework Programme (FP7/2007-2013) under grant agreement n°287634

## Acknowledgement

This report forms part of the deliverables from a project called "FoodMicroSystems" which has received funding from the European Union's Seventh Framework Programme FP7/2007-2013 under grant agreement n° 287634. The Community is not responsible for any use that might be made of the content of this publication.

FoodMicroSystems aims at initiating the implementation of microsystems & smart miniaturised systems in the food sector by improving cooperation between suppliers and users of microsystems for food/beverage quality and safety.

The project runs from September 2011 to August 2013, it involves nine partners and is coordinated by ACTIA (Association de Coordination Technique pour l'Industrie Agro Alimentaire, France). More information on the project can be found at <http://www.foodmicrosystems.eu>.

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## Executive Summary

The present document describes the roadmapping process made for the development and use of microsystems (MST) in the beverage sector. This sector has a capital importance within the Food industry in Europe and has been selected and studied in detail within the FoodMicroSystems project.

Food and beverage applications provide an excellent arena for developing sensing/actuating platforms, and such applications will get in turn profitable advantages from those systems in terms of optimised production control loops, costs savings, and improved quality and safety. Both sectors are main exporters and world leaders but are threatened by new producer countries from outside Europe that have entered strongly in the global market. Microsystems seem good candidates to be accepted by beverage manufacturers, provided that may help on developing realistic alternatives or complementary to existing analysis methods based on the use of expensive laboratory equipment.

During the discussions carried out for implementing this report, it has been concluded by experts that the improvement of the process control with new microsensor-based devices can ensure not only the optimisation of the process from economic and environmental points of view, but also the quality and safety of the final product. Therefore, wine and beer elaborators identified the main critical points for which a set of product attributes or process parameters are capital to be monitored: receiving raw materials, fermentation (which is the most important stage for making wine and beer), maturation, bottling, logistics and transportation.

Roadmap results show that chemical and biochemical detection devices and sub-systems may improve the performances of current miniaturised e-nose or e-tongue systems and that some implementations are possible in a short time frame (three years), since a variety of basic components of microsystems already exist and only need to meet the needs of the application. In other cases, it is necessary to implement new components and subsystems from scratch and, therefore, results can only be expected at mid to long-term (five to ten years). Finally, the winemakers and brewers have also shown interest in microsystems for addressing not yet solved analytical challenges. In these cases microsystems experts have evaluated which sensor families would be most useful and which would be the technological issues to be addressed. This information was also discussed with instrumentation developers.

The priorities of beverage elaborators in having new measurement systems do not always coincide in time with the expected availability of some of the proposed microsystems. However, even in these cases, the priority given by the end users and the information obtained is also useful for MST and instrumentation developers because it can help on prioritising the MST R&D activities because of their market demands, which at the same time will drive funding authorities when defining what and when to fund in the MicroNanoBioSystems (MNBS) arena.

## 1 Introduction

This FoodMicroSystems report intends to combine the needs of the beverage sector (but mainly dedicated to wine and beer) with the opportunities that microsystems technologies will be able to offer for this sector within a timeframe of about three to ten years.

The “Microsystems for the beverage sector roadmap” is one of a series of three “application driven technology roadmaps” that will lay the foundation for future collaboration between the food/beverage and microsystems sectors, support strategy decisions of research organisations and companies, and supply investors and funding bodies with guidelines to support their investment decisions.

The roadmaps are application driven, whereby trends for future application areas and products are identified and their “in-service” dates forecasted. In this context, the underlying microsystems technologies provide the basis of the secondary layers constituting the roadmaps. We have identified significant overlaps in microsystems needs within the three application-driven food roadmaps, thus it was decided to integrate this secondary layer of microsystems roadmaps into a separate report D4.5, jointly for all application roadmaps (dairy, meat, and beverage).

The benefits of smart miniaturised systems based on microsystems (MST) will be discussed in general for the beverage sector, but particularly and with more detail for the winemaking sector, that will be taken as a case study, but also extended to the beer production, when relevant. This is because of several reasons. Many of them are not exclusive of the wine/beer sectors but all of them concur in them. The first is that wine offers one of the richest sensing scenarios of all food/beverages: it is the more extended in space, time and types of substrates (from growing grapes in vineyards to processing must and getting final wine in cellars, a year of continuous processing is needed). Wine merits not only as a natural product with positive social, cultural and environmental effects, but also as a highly relevant European industry in terms of revenues and employment. In addition, this sector is atomised in all sorts of company sizes, every type of which may find a different advantage in the possibilities offered by the implementation of analytical instruments, thus providing an excellent test-bed for end users Smart Systems acceptance.

Wine is a high added value product and can afford more easily newer technology. Moreover, worldwide overproduction has led to increased competition and a growing demand for quality wine at the right price. New world producers are challenging the leading role of the winemaking European industry. Improving quality and optimising the production process have become key objectives for European producers seeking to keep their competitive edge, and this must be achieved by the introduction of new technologies in this industry.

Wine is a high added value product of capital importance for the European Economy. The position of the EU in the world wine sector is of pre-eminence and still healthy. However, its dominance is declining slightly year after year because worldwide overproduction has led to increased competition and a growing demand for quality wine at the right price. It is foreseen that new technologies for ensuring and improving wine production quality, safety, and reduction of process costs may help on reverting such tendency. Optimising the production process has become key objectives for producers seeking to keep their competitive edge while addressing sustainability, too.

For the beer sector most of the above mentioned reasons also apply, and in addition it brings the advantage of being a larger sector in terms of production, with some specificities due to the normal shorter processing time, but also because of the different size of the companies dealing with the beer production compared to the wine subsector. These specificities will be treated at each stage when they become relevant for the discussion.

In the case of non-alcoholic beverages, the variety of products and elaboration processes is even much more important, but the sensing demands are common in most cases. In the following paragraphs such demands are highlighted.

## 1.1 Importance of the beverage sector in the food & drink EU economy



Within the food and drink sector, the beverage chapter is of great significance as it can be observed in figure 1.1: while only amounting 8% of the companies, it represents a 15% turnover, 10% employment and 18% added value of the whole sector. Meat is the first product in importance in turnover terms, and bakery & farinaceous products ranks first in the other categories. Tea and Coffee as part of the “various food products” category (which also includes sugar, prepared meals and baby food) stands for an additional 1% of the total shares. In addition, beverages sector was in the first position in terms of EU food exports shares with 29% in 2010<sup>1</sup>.

<sup>1</sup> <http://www.fooddrinkeurope.eu/publication/fooddrinkeurope-competitiveness-report-2011/>

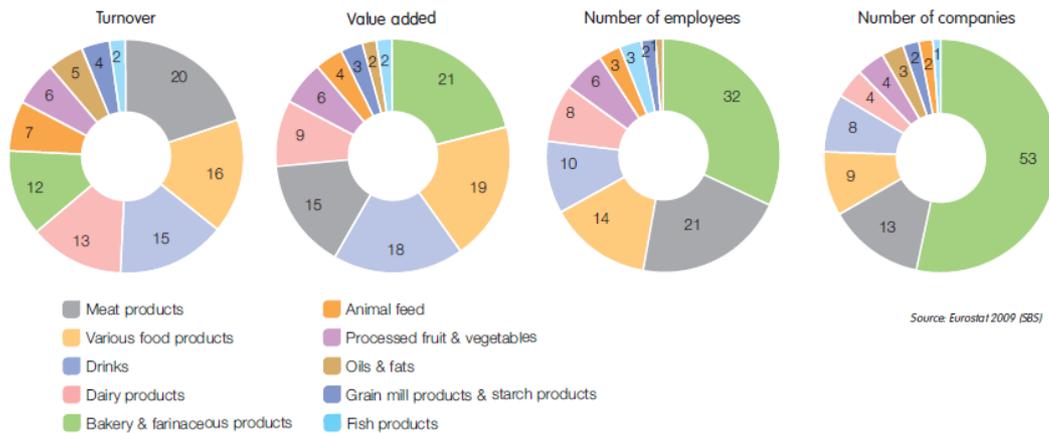


Figure 1.1: Relative importance of the different sectors of the EU food and beverage industry (last available data in FoodDrinkEurope 2011 report)

Within beverages, the sector of alcoholic drinks is one of the most important as compared to sodas, water, juices, etc... The alcoholic beverages group the type of drinks that contain ethanol or ethyl alcohol and are the result of the natural process of fermentation of fruits, grains, vegetables, plant matter, and even dairy products. They show some specific and important characteristics: they are of high added value, there is an economic impact for Europe in terms of imports and exports, there are socio-cultural aspects related to their consumption, and last but not least, their processing is much longer and complex. In this domain, the three main subsectors are wine, beer and distilled spirits. The importance of each is shown in figure 1.2.

### alcoholic drink production (Mhl)

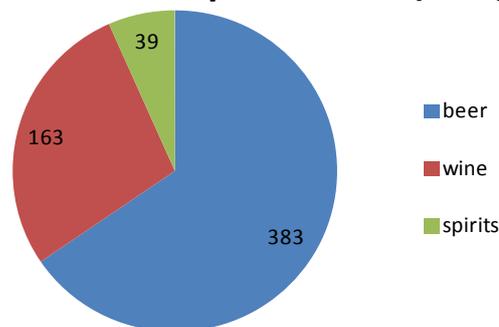


Figure 1.2: Relative importance of the wine, beer and spirits in EU alcoholic beverage production (data from 2010, different sources).

### Economic importance of the wine sector in Europe



The structure of the wine sector is very different compared to the one of the meat or dairy industry due to the importance of small businesses. Two types of business models coexist: independent wine growers

who cultivate their grapes, produce and commercialize their wine and larger companies (often cooperatives) that collect the grapes from vine growers and transform them into wines. In the EU there are around 600.000 wine growers producing quality wines and more than 1.75 million of farmers involved in the production of “other wines”.

The EU is the largest wine producer in the world, as well as the world’s leading exporter of wine products, and the sector contributes with around 15bn annually to the EU economy<sup>2</sup>. The wine sector’s importance to the European economy should not be counted merely in monetary terms, however. The sector permeates many levels of European life, contributing significantly to society in socio-economic, employment, environmental and societal terms as will be discussed afterwards.

Traditionally, most European cultures consider wine a refined choice but with a very important added value and a pivotal role of wine in European tradition, social tissue and its impressive numbers in European economy. This wine concept as an enduring cultural symbol of European living has been exported with success to the rest of the world. With so much success, in fact, that an increasing number of New World producers are claiming a bigger part of the world wine market share, arising competitiveness and sustainability concerns in the European wine sector.

The European Union is the world’s leading producer and consumer of wine and:

- In 2011<sup>3,4</sup>, roughly 50% of the 7.6 MHa vineyard surface around the world was located in Europe. Spain, France and Italy led the ranks followed by Turkey, China, USA and Iran.
- 60% of the 265Mhl of wine produced globally had its origin in Europe. Italy, France and Spain were leaders followed by USA, Argentina, China, Australia, South Africa and Chile.
- Regarding consumption, EU accounted for the 50% of the 242Mhl consumed worldwide. France, then USA, and then Italy and Germany are the four main wine consumers.

The EU is also the first trader in the wine market:

- It is the first exporter of the world (70%) with again Italy, Spain and France leading such international trade, Australia and Chile following.
- On the other hand, because of the climate and environmental requirements for grape growing it is clear that not all countries can match consumption and

<sup>2</sup> From the Report “Towards a Sustainable European Wine Sector”. July 2007.  
[http://ec.europa.eu/agriculture/publi/fact/wine/072007\\_en.pdf](http://ec.europa.eu/agriculture/publi/fact/wine/072007_en.pdf)

<sup>3</sup> <http://www.winesfromspain.com/icex/cma/contentTypes/common/records/viewDocument/0,,00.bin?doc=4626818>

<sup>4</sup> *Statistical Report on world vitiviniculture 2012 in www.oiv.int*

production and, thus, EU is also the first importer region (70.5%), with Germany and UK being the first world importers followed by USA and Russia.

- While in recent years, the volume of wine imports into the EU has been growing, the EU remains a net wine exporter with a net surplus of ca 3-5 billion € per year.

The wine market is very dynamic and exports play a major role in the development of the sector: the demand for wine is expected to increase by 10% between 2012 and 2016, due to a spectacular increase of the consumption in Asia (+40% in China). In this scenario, the position of the EU in the world wine sector is of pre-eminence and still healthy. However, its dominance is declining slightly year after year in almost every front.

### Economic importance of the beer sector in Europe



Beer as wine is an enjoyable beverage which brings pleasure and social interaction to many people. When consumed in moderation – as done by the vast majority – beer and wine can be part of an adult’s balanced and healthy lifestyle. Beer is the world’s oldest and most widely consumed alcoholic beverage (with relatively low alcoholic content) and the third most popular drink overall after water and tea. Beer has been produced for thousands of years, by the brewing and fermentation of starches, mainly derived from cereal grains-most commonly malted barley.

Beer processing also relies on the control of the fermentation process. In fact, **beer production is much less seasonal than wine production and fermentation takes place all the year around.** Moreover, most of the different types of beers (lager, ale, stout...) differentiate mainly because of the fermentation phase. World beer sales were around 1,300 million hectolitres in 2010, declining or, at best, remained static over the past few years. This has been due to a combination of continued maturation of the market and intensified competition from wine and spirits. However, the beer sector is still a large contributor to the European economy. According to the study of Ernst and Young<sup>5</sup>:

- The production of beer was of 383 million hectolitres in 2010 in the European Union (EU). Beer consumption in the same period was of 343 million hl with a sales value of around 106 billion €.
- Both production and consumption has slightly decreased in the last years (6% and 8% respectively since 2008) not only driven by the recent global economic downturn, but also because the decreasing consumption trend started a number of years ago and is expected to continue, especially for Premium Beers.

<sup>5</sup> [http://www.brewersofeurope.org/docs/flipping\\_books/contribution\\_report\\_2011/index.html](http://www.brewersofeurope.org/docs/flipping_books/contribution_report_2011/index.html)

It is important to know that in the European Union, breweries are mainly micro and small breweries. Nevertheless, the EU remains one of the major beer producing territories in the world, as compared with Russia (101 million hl), Brazil (122 million hl), the United States (207 million hl) and China (466 million hl)<sup>6</sup>. It was only in 2008 when China passed the EU as the biggest beer producer in the world. European beer brands are sold worldwide, either brewed in Europe and exported, or produced abroad mainly by subsidiaries of the larger European brewing companies or under license by other brewers in those countries.

Around 3.600 were active in Europe in 2010. Germany has the largest number of breweries (1,325 in 2010). Six other Member States have more than 100 breweries each: United Kingdom (824), Italy (353), Austria (172), the Czech Republic (145), Belgium (135), Denmark (120) and Poland (103). It is estimated that around 70% of the European breweries are microbreweries (small producers of beer that serve local or regional markets with maximum production around 10,000 hl).

The main European beer producers are Germany (106 million hl), UK (58 million hl), Spain (31 million hl), Poland (27 million hl), The Netherlands (25 million hl), Czech Republic (19 million hl), France (18 million hl), Belgium (16 million hl), Italy (14 million hl) and Denmark (8,5 million hl).

Despite production in each country is usually tuned to the local consumption level, all of the 27 Member States also import beer to a greater or lesser extent. In 2010 the total import volume for the 27 countries was 43 million hectolitres of beer, and of 73 million hectolitres of exports. Most export partners are located within Europe. The most important export partner outside Europe is the United States. Exports are especially important for the Netherlands, Belgium, Denmark and Ireland, where the percentage of exports compared to total domestic beer production is high (between 50 and 60%).

## **Food/wine production and climate change**

For many key parameters, the climate system is already moving beyond the patterns of natural variability within which societies and economies have developed and thrived. Water is one of those threatened resources. A summary of threats in Europe<sup>7</sup> is presented in figure 1.3.

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<sup>6</sup> Canadean Global Beer Trends 2010.

<sup>7</sup> [http://ec.europa.eu/agriculture/climate\\_change/workdoc2009\\_en.pdf](http://ec.europa.eu/agriculture/climate_change/workdoc2009_en.pdf)

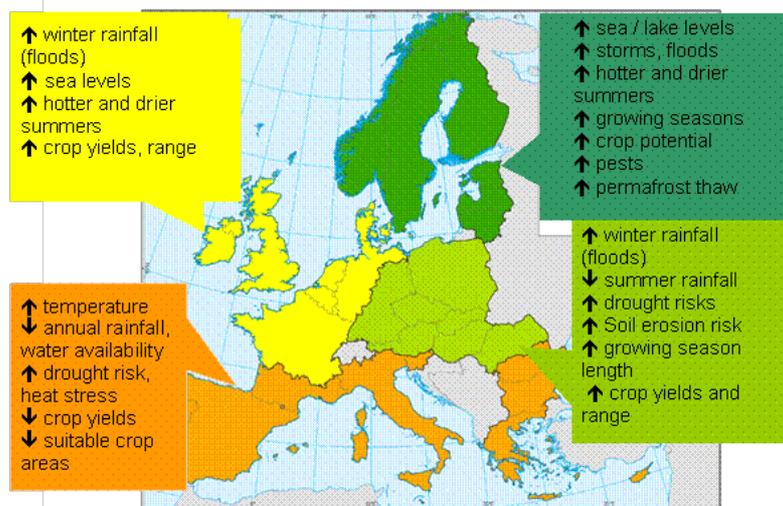


Figure 1.3: Projected agrofood impacts from climate change in different EU regions

Socio-economic pressures over the next several decades will lead to increased competition over decreasing freshwater resources between population needs and industrial/agricultural needs, potentially reducing the availability and quality of water resources for the later. As a consequence, the European Commission adopted a policy paper on the challenge of climate change for European agriculture and rural areas<sup>8</sup>. The paper examines adaptation needs, and explores possible orientations within the agricultural sector for future action. Using water more efficiently by reducing water consumption and losses, and recycling or storing water is identified as a key issue, which food/beverage industry will have to address mandatorily in the near future.

### Environmental aspects in food/beverage production: water quality monitoring and savings during processing



Despite most of the water consumption (up to 70% of fresh water) for food and beverages is in agriculture, the issues related to water savings are also very important for the food industry during production. Due to the variety and high number of SMEs in the sector available data on water use in Europe in the sector is incomplete, but estimates<sup>9</sup> from various national food and drink industries suggest that this sector is responsible for a share of between 8% and 15% of industrial water use, which would represent between 1% to 1.8% of the overall fresh water use in Europe.

In the food and drink manufacturing sector water may be an ingredient (for non-alcoholic drinks and some alcoholic drinks) or a final product (bottled water).

<sup>8</sup> [http://ec.europa.eu/agriculture/climate\\_change/workdoc2009\\_en.pdf](http://ec.europa.eu/agriculture/climate_change/workdoc2009_en.pdf)

<sup>9</sup> Managing environmental sustainability in the European Food and Drink Industries. 2<sup>nd</sup> Edition. 2008. CIAA

Moreover, water is an indispensable element in many processing steps, such as washing, boiling, steaming, cooling and cleaning. Beer is an example of alcoholic drink in which water is an ingredient, while wine it is an example in which water it is not. Water management and waste disposal have become a significant cost factor and an important aspect in the running of a winery and a brewery operation. Every producer tries to keep waste disposal costs low whereas the legislation imposed for waste disposal by the authorities becomes more stringent. Water consumption in a production site is not only an economic parameter but also a tool to determine its process performance in comparison with other sites. Furthermore, the position of wine and beer as a natural product leads the producers to pay attention to their marketing image and to take waste consumption and treatment into account.

For the specific case of wine and beer manufacturing, an average amount of 5 and 7 litres of water per litre of manufactured product is well agreed by wineries and breweries, respectively. This amount is already lower than some decades ago. This means that there is a trend of water saving, not only because of water cost increases but also because of the environmental impact of the discharges of potentially dirty water. There are technical limits for decreasing the usage of clean water in the food industry. For example, the re-use of water from on-site cleaning stations is regulated by Regulation (EC) No 853/2004 on the hygiene of foodstuffs, stipulating that recycled water used in processing or as an ingredient has to be of the same standard as clean drinking water. But even within those technical limits there is still a lot of room for improvement.

Improving water efficiency in cleaning raw material (grapes, cereals...), pipelines and tanks during processing, may be done by the introduction of new technologies, which are capital intensive, but also by controlling the quality of the effluents during cleanings. A closed loop control based on systems sensing water quality is just a necessary element in this process. In addition, wine and beer production is known to produce high amounts of wastewater with sometimes extremely high organic loads (COD 2.500-67.000 mg/L). This leads to shock loads to municipal wastewater treatment plants or to serious impacts on rivers and lakes. Also the control of discharges of wastewater is mandatory by regulations if the processing plant does not have a wastewater recycling plant (which is the case of the great majority of SME producers, as a water treatment plant may cost up to several million-Euros for big production plants, and only the biggest manufacturers can afford them). A system fingerprinting wastewater quality will be certainly decisive in the water reuse/disposal strategy.

## 1.2 Sensing demands of the beverage sector



The beverage industry, mainly composed of the subsectors of wine, beer, spirits, soft and mineral waters, cannot be studied in a homogeneous way since the products covered are very different. The complexity and processing time, the needs of control of the quality and safety of the products and of the manufacturing processes are also very different depending on the sector we are dealing with. However, in all cases some generic needs are identified, that require the use of sensing instruments for the:

- Determination of the quality and safety of raw materials and final product.
- Monitoring and automation of the production process to ensure the safety and quality.
- Ecologically processing for reducing the energy and water consumption.

The work of the winemakers and producers of beers, spirits and other beverages is therefore to define a set of parameters, attributes of the quality and safety of the product and to follow analytical procedures (usually in specialized laboratories) to determine them and, thus, to ensure adequate product processing and final quality. In the beverage industry the aroma, taste and colour are the most important quality attributes and many of the analysis and measurements done are related to them.

However, the beverage processing techniques, especially in the wine industry, are usually very traditional even for the major brands and the oenologist are often made in a manual form, several times a day. The winemaking process (graphically shown in figure 1.1.1) depends on the type of wine that is being developed (white wine, rose, red, table, vintage, sweet, dry, young, old, still, sparkling,...) but has common requirements related to the:

- discrimination and classification of the raw material (grapes, must, wine) according to their origin, variety, quality, phenolic maturity level, plant health, tampering, ...
- quality of containers: bottles, cans, barrels, corks, etc... : aromatic attributes, cleanness and sanitary condition.
- monitoring control parameters (temperature, density, colour, O<sub>2</sub>, SO<sub>2</sub>, pH ...) during different production processes (fermentation, maturity...) and its evolution over time.
- yeast cell growth monitoring
- monitoring of quality parameters of wine and their evolution throughout the process: phenols, tannins, volatile antioxidant capacity, sugars, ethanol...

- detection of defects due to the process indicators or potential security issues: acidity, astringency, sulphites, allergens, pathogens, toxins, anisole, pesticides ...
- optimisation of production processes for wine or grapes from different sources.
- monitoring the bottling process and the wine attributes in the bottle
- final product quality: taste, aroma, alcohol content, temporal stability ...
- control of ambient conditions (humidity, temperature, light...) during the logistics and transport
- control of water quality used during the process. Control of effluents, chlorine, heavy metals, organic contents...

## The wine food chain

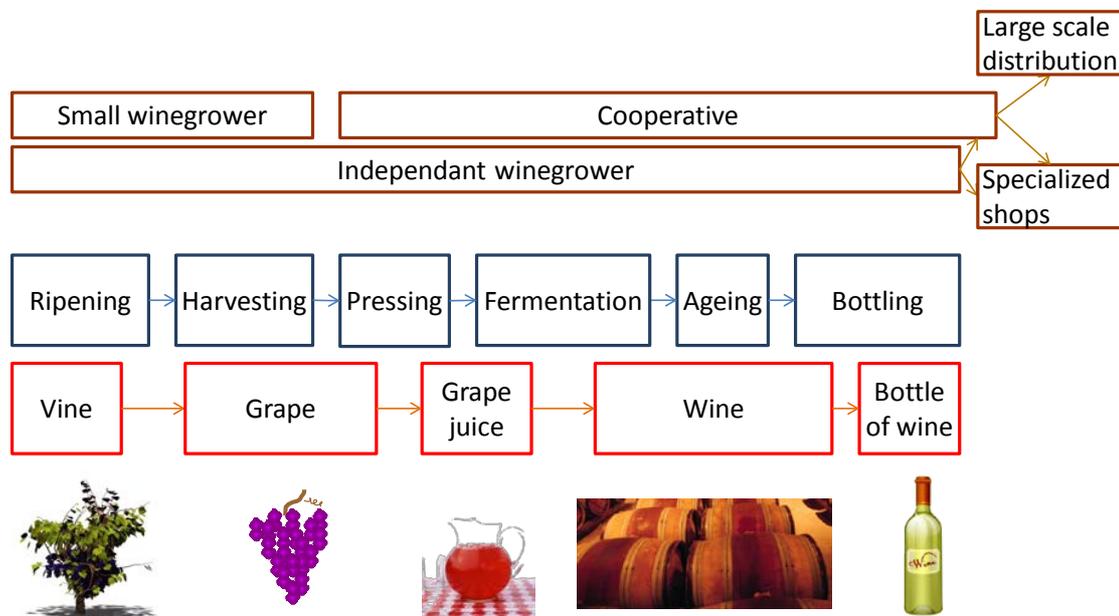


Figure 1.4. Graphical description of the wine food chain as a case example of the processing in the beverage sector.

These requirements are also important for the other beverage subsectors. For instance, the beer sector shares many of the wine attributes, processing steps (including alcoholic fermentation as the main one) and monitoring needs. As for wine, there are also many types of beers that are obtained following different processes: pilsen, lager, stout, ale, wheat, malt, dark, pale, white, non-alcoholic beers, ..., but consumers expect that, unlike the case of wine, the organoleptic properties of the beers of the different brands remain stable over time. Thus, discrimination among beers and prevention of frauds are of vital importance, together with other specificities like:

- Discrimination of types of alcoholic and non-alcoholic beers, alcohol contents
- Determination and monitoring of the quality of the specific attributes of beers: bitterness, sourness, sweetness, rheological properties, colour, taste, body, intensity and duration ...
- Changes on the quality of beer depending on type of packaging (cans, bottles ...).
- Effects on the safety for consumers: determination of traces of thiamine, gluten and protein contents, sulphur dioxide, metal can corrosion.
- Traceability and logistics outside and inside (for major brands) the production plants.

For the case of non-alcoholic drinks there are also specific attributes and parameters for the various types of them (a big variety of mineral waters, sodas, juices, teas, ...) that, unlike wines and beers, are not elaborated based on the alcoholic fermentation process and on the aging during long periods, but on much more industrial and faster processes. For these types of beverages, we can identify again some specific characteristics and control parameters to be considered, such as:

- Authentication to geographical origin of coffees and teas. Classification and quality control through the determination of volatiles of leaves and grains.
- Quality attributes: caffeine and phenols contents, antioxidant effect of infusions.
- Vitamin contents of fruit juices and percentage of fruit. Shelf-life determination.
- Colour and sweetness and CO<sub>2</sub> contents of soda and coke based beverages. Rheological properties
- Processing changes of quality: effects of stabilization processes (UHT, pressure, electric fields ...) in colour, taste and flavour of beverages.
- Safety issues: Foodborne bacteria and pesticide traces in fruit juices, fungi contamination of green coffee grains...
- Quality of bottled water, carbonates, ions, ...

In summary, the entire beverage sub-sectors show complex processes that require monitoring procedures and instruments. Up to now, laboratory instruments and different analysis methods and complex devices have been developed in recent decades to meet the above needs. The main techniques are: Surface Plasmon Resonance, Fourier Transform Infrared, Ultra Violet and Mass Spectrometry, Spectrophotometry, Spectrofluorimetry, High Performance Liquid Chromatography, Gas Chromatography, Turbidity, Density, etc... Normally instruments based on such techniques are very precise and the main ones are also of very high cost and are not available in most of the laboratories of beverage elaborators. However, in many cases these techniques suffer from a slow response and cannot be used on-line.

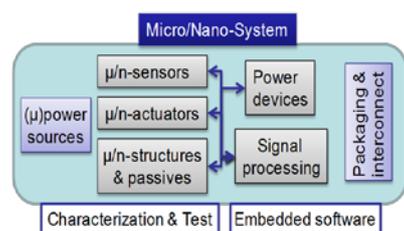
A real advantage would be to develop fast and reliable and fully automatic systems that would allow simpler, faster and cost effective sample processing and sensing.

Especially in the last decade an important effort has been done at R&D and also at industrial levels in order to develop novel sensors miniaturised sensors that are the basis of portable systems and that help on the automatisisation of the monitoring of beverage goods. Research on physical, chemical and biochemical sensors, adapted to the food sector requirements has been done in order to evaluate their chances for replacing complex systems. Most of them were based on non-silicon device and technologies that have also been part of complex but miniaturised systems.

For instance, e-nose and e-tongue based on chemical and biochemical sensing devices are well known systems that, however, have not yet achieved the commercial success, probably because of their cost, lack of selectivity and stability and not good enough sensors. In most cases that have been tested with success as qualitative systems but not as quantitative devices with good performances and reliability. However, these systems may also improve as soon as they are made more specific to the application, thanks to the development of on purpose-developed sensors, more performing too and with better sampling and concentrating stages.

Here is where microsystems and microfabricated sensors may play a role, both at the transducer and at the systems levels, thanks to their inherent performances. The present roadmap intends to address which are the improvements of micro and nano fabricated sensors, transducers and microsystems in order to be commercially successful in the next decade. The roadmap exercise also takes into account not only the technological requirements but also the priorities given by the end users and machinery developers for the beverage sector.

### 1.3 Microsystems for the Beverage Sector. Vision until 2020 and beyond.



Microsystems (MST) are electronic systems that are usually composed of a combination of sensing and or actuating devices, signal processing and communication, and power supply subsystems that together bring an added value in terms of smartness. They are also known as ICT/MST or MST

or MEMS (MicroElectroMechanicalSystems) or integrated systems or Smart Systems, according to world region of use. As for many other applications already established in the market (transport, health, environment...) they may provide information about the food and beverage products content or their change of properties during their processing and life. The systems involve at least one component built by Micro or Nanotechnologies (MNT) following in most cases microelectronics based batch fabrication process (see figure 1.5). Additives in food are not in the scope of microsystems.



*Figure 1.5. An overview of micro-technology batch processing of sensors and micro-systems before they are packaged according to a final application*

In the above described beverage scenario, new sensors and microsystems may play a crucial role as they may bring to the beverage sector, well known advantages like:

- miniaturization
- fast response
- cost reduction
- in-line and on-line use
- continuous electronic reading and smart communication

A variety of sensors, and systems are available at research level and some of them also have reached the market, that are available to the beverage sector, but most of them are not yet based on microsystems technologies. Thus, the aim of this roadmap is to identify key quality and safety attributes of wine during processing and the technological solutions that can be addressed in the future with microsystems delivering an improved approach compared to laboratory analysis.

## **Vision beyond 2020**

Microsystems also aim to provide solutions beyond 2020 and, in this case, more speculative drivers for the future can be found: developing systems smarter, smaller, cheaper and that consume as low energy as possible. These systems will have to be multiparametric and will have to allow real time monitoring of the maximum number of quality indicators and/or defects and provide the fastest response during processing and, thus allowing the generation of a corrective action as quickly and efficiently as possible.

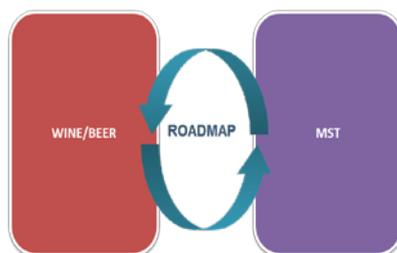
The systems will have to be usable in various critical points and, therefore, will have to be solutions to the many needs that we can encounter during all processes. So, using the example of wine and beer making, this roadmap identifies as long-term vision systems solutions that will implement the following paradigms:

- **The intelligent weighbridge and reception point:** where all materials (grape, crop and others necessary for processing) are evaluated in a very short time in terms of sensory evaluation, quality and safety.
- **The intelligent tank:** where fermentation is fully smart monitored, both controlling at the processing parameters in the tank and also the variation of the quality attributes of most when it is changing to wine.
- **The intelligent barrel:** where wine processing and product quality and safety controlled during ageing
- **The intelligent bottles and corks:** where wine is continuously controlled with non-contact systems during the bottling process and stabilisation.
- **The intelligent logistics:** where smart sensing and communication processes, RFID's and labels allow the tracking of the quality of the product until it reaches the consumer.

In all these scenarios it is expected that new families of microsystems will be developed at medium – long term that will comply with the following requirements coming from a visionary scenario of maximum performances and simplicity of use for the winemakers:

- Maximum automation. No need for calibration. In situ data treatment. Better data processing and easy training approaches.
- Multiparametric systems for qualitative and quantitative determination. Maximum integration of sensors at minimum cost.
- Minimum sampling, non-invasive, non-contact (from outside the packaging, if possible) measurements.
- Combination of direct and indirect measurement strategies for maximum simplification.
- Combination of improved (bio-) sensing materials, bio-recognition techniques, with electronics, improved signal processing, RFID's...
- Zero power or ultra low power solutions. Energy scavenging.
- Moving systems from quality control of end products to monitoring during the full process. Wider application fields.

## 1.4 Roadmap methodology



The application-led roadmap has been developed for forecasting industry's needs and research challenges. However, predicting the future is difficult and, thus, a big effort has been put for getting the points of view of different types players of the beverage sector. Because of its complete and complex process that covers the main needs in

terms of analytical tools, wine has been taken as the backbone of this study and, when relevant, additional considerations especially for the beer sector have been taken into account, too.

The objective is to develop “wine and beer application driven technology roadmaps” for the microsystems sector that can draw the foundations for new analytical systems. The roadmap process is being carried out in different steps being the main ones, the:

- a) Identification of main wine quality attributes and parameters during processing, and their priorities.
- b) Identification of potential sensor and microsystems solutions for determining such attributes.
- c) Defining the technological steps required for developing such sensors and systems if they are not yet available, and defining the degree of its technological complexity.

The information for starting the discussions for defining the roadmaps has been mainly obtained from strategic agendas and other working documents of food and beverage associations and from “brainstorming” sessions in open events especially organised by the FoodMicroSystems project with winemakers, oenologists, analytical instrumentation companies and Micro and Nanosystems experts mainly from Spain, France and Italy. Visits to winemakers and brewers in their factories have also been done and, finally, preliminary roadmap results have been presented and discussed in the World Conference of OIV (International Organisation of Wine) held in Bucharest in June 2013.

The results of the analysis of points a) and b) give as result a set of diagrams that graphically show the priority in time (three, five and ten years from now) of each attribute or indicator according to the wine producers point of view, and the associated sensor and/or system solution and its degree of complexity, according to the experience of MST and instrumentation developers. Point c) will be elaborated later for the more challenging and critical set of systems identified.

In section 3 the graphical outcomes of the FoodMicroSystems study of the main wine process steps (also including specificities for the beer sector) are presented.

## 2 Main drivers for innovation

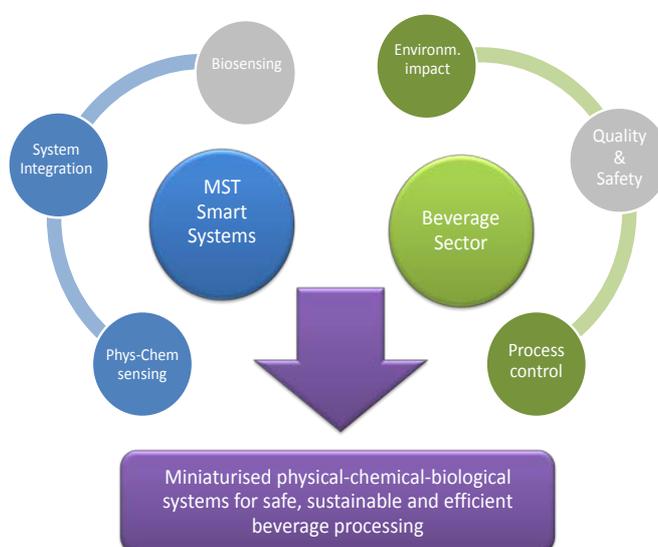
The Eurofound<sup>10</sup> report on trends and drivers of change in the food and beverage production industry already stated some years ago that automation of plants and processes and new technologies are paving the way for more effective forms of production and the development of new products. Among the key technologies identified in the Strategic Research Agendas of the sector (i.e. from FoodForLife and OIV international organisations and also from the Spanish Technological Platforms of

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<sup>10</sup> [www.eurofound.europa.eu/pubdocs/2006/37/en/1/ef0637en.pdf](http://www.eurofound.europa.eu/pubdocs/2006/37/en/1/ef0637en.pdf)

Wine PTV and Innovi) are biotechnology, information and communication technology (ICT), radio frequency identification (RFID), robotics, and sensor and microsystems (MST) technologies. These MST technologies that allow fabricating electronic based sensors and components with dimensions in the range of microns and nanometers may be the backbone of new tools, devices, methods or services that can provide relevant information to orient the technical decisions of wine producers, overcoming the costly and long-time current laboratory analysis.

Figure 2.1 graphically summarises the motivation of FoodMicroSystems Project, which is to combine expertise from the microsystems and the beverage sectors in order to help on defining a new generation of analysis tools that may give added value to the current methodologies used in the sector.



*Figure 2.1: Main drivers for innovation: Mixing beverage sector requirements with MST potentialities.*

The impact of microsystems will depend on the value that beverage producers concede to the fact of having key information at critical check points in their process, supplementing their knowledge and experience, helping them to make the right decisions in pursuit of a safe product and of quality. Figure also refers to process points, in which sensors and microsystems can play a role, related to reduction of consumption of water. But process control is not a must for optimising the production processes in terms of efficiency. Monitoring of all the steps of the production chain is also part of the optimisation of other key drivers for the beverage sector that, as expected, are related to the quality and safety of their products.

Figure 2.2 summarises the preliminary views of the wine sector (mostly applicable to the beer sector, too) in terms of global drivers, industry priorities and how they can be connected to the need of developing new microsystems technological solutions. The priorities for these drivers are not absolute but connected to the availability of the technologies and on the specificities of the products. This means for instance

that the safety driver which in principle should be of high priority for any kind of beverage, it is considered of lower priority by the wine and beer sector as, alcoholic products are more inherently safe products compared to other types of beverages and, thus, other priorities like quality of the product and cost efficiency are taken as a better drivers for new microsystems developments.

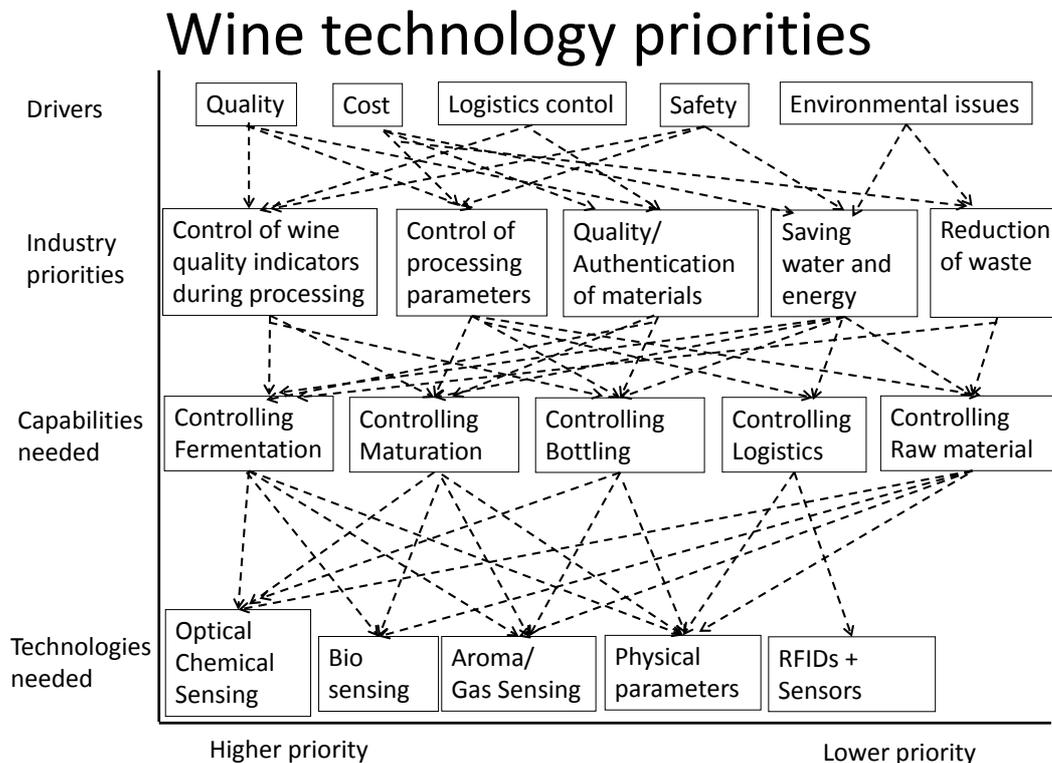


Figure 2.2: Drivers, and Product needs that impact the microsystems technological developments for the wine sector

Starting from this planning, the question is “in which areas of the supply chain are”:

- Problems to be solved? ...maybe through microsystems?
- New opportunities for microsystems?

## 2.1 The need for testing & measuring

For answering these questions other preliminary questions have to be analysed:

- where we have to measure?
- what we have to measure in each place?
- which systems are available now?
- what microsystems can do?
- what types of systems have to be developed and are of more interest?
- which technological steps have to be proposed in the next years to achieve the needed systems at the right time?

All these questions are addressed in Deliverables Del 4.4 and Del 4.5.

## Where to measure?

Figure 2.3 summarises the supply chain of wine (as the main example for the alcoholic beverage sector), but that en general also defines the beer process.

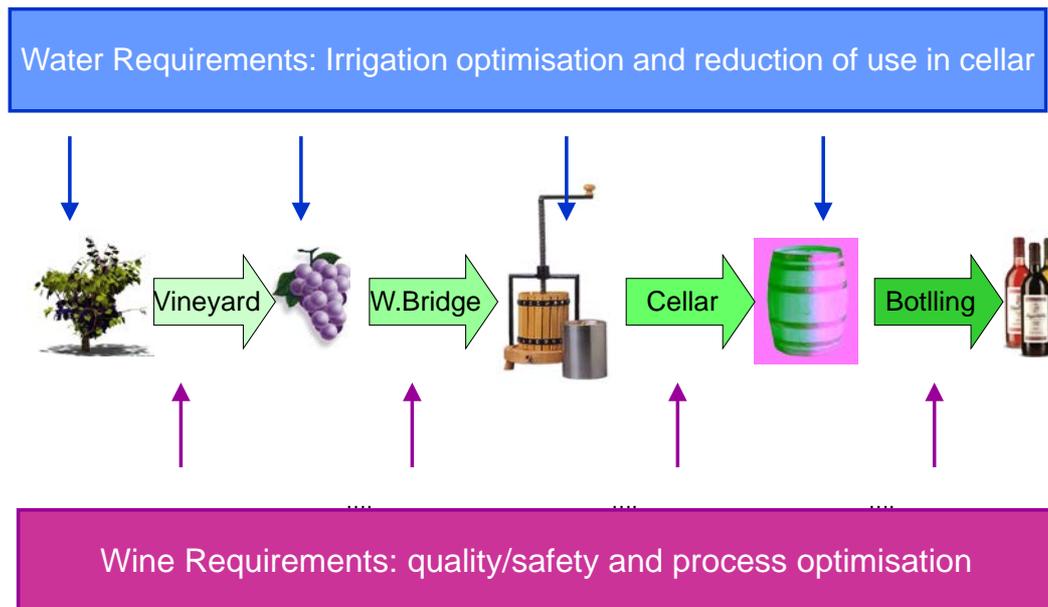


Figure 2.3: The cellar scenario in relation to the 'wine' and 'water in winemaking' chain, showing in arrows the main processing steps (fermentation, maturation, bottling, cleaning,)

From the figure we see that there is a need of new sensors and microsystems at each point for which a material has to be analysed or a process has to be monitored during time. Five main scenarios (which some of them can be split in other sub-scenarios) were defined for the next discussion:

- Raw Materials reception
- Fermentation in tank and preparation of the fermentation promoters
- Maturation and ageing in barrel, tirage (for champagnes)
- Bottling, 2nd fermentation (for champagnes), stabilisation, logistics
- Other on-line and off-line controls

In these scenarios, many of the key product indicators or process parameters are common for both wine and beer. However, beer is produced in higher amounts, it uses to be of lower cost and matured products are not so common compared to wine. Thus, some specificities will be highlighted when necessary in the next chapters.

## What to measure?

Table 2.1 presents the main parameters of interest of the wine and beer sectors that is a summary of the preliminary discussions held with key representatives of the sector, including wine and champagne and cava producers, in order to take into account the differences in the process. These parameters come from open discussions held in preparatory meetings and from visits to important winemakers and they were taken as points for discussion in the specific face to face meetings specifically held in a second phase for defining the roadmaps contents, as presented in the next chapter.

It has to be stated that not for all the indicators presented in the table, it makes sense to use microsystems, as the acceptable cost of new testing devices and methods may not be economically attractive if the systems are not deployed in large markets. However, these considerations are also part of the roadmapping exercise and are another challenge for the microsystems developers. Another decisive acceptance factor for proposing new test methods is to shorten the time to result, while achieving the necessary sensitivity, selectivity, accuracy and quality of results.

									
Goods Reception	Fermentation Promoter	Fermentation	Tirage	Ageing and Maturation in Barrell	2nd Fermentation. Ageing in Bottle	Desgorge	Bottling	Logistics	Other (cleaning, etc...)
Colour	Density (Sugar)	Density (Sugar)	Tartaric Stabilisation tartárica (Potassium, Calcium, ...)	Oxygen O2	Oxygen O2	Sugars	Detection of foreign particles	Traceability with RFID with sensors	pH control
Phenolic maturity	Temperature	hydrogen sulfide	Electric Conductivity	Redox Potential	Redox Potential	Sulphur dioxide SO2	Oxygen O2	Temperature	DQO control
Richness Aromatic	Viable yeasts (on line)	Mercaptanes	pH	pH	Turbidity	pH	Sulphur dioxide SO2	Humidity	Chlorine contents
Contaminating Microorganisms		Viable yeasts		Sulphur dioxide SO2	Sugars (Champagne)	Acidity	Pressure	Light	
Gluconic Acid (Sanitary status of grapes)		Lactic Bacteria			Pressure (Champagne)		Turbidity	Intelligent inks	
Other Horizontal applications: <ul style="list-style-type: none"> <li>• Identification and authentication of raw material and finished product</li> <li>• Cork Quality (TCA)</li> <li>• Multiresidue detection / allergens</li> <li>• Microbiological Control (Brettanomyces)</li> <li>• Organoleptic objectification</li> </ul>									

*Table 2.1. Summary of the main parameters and attributes of wine during processing obtained from the first “brainstorming” discussions with wine makers. At this stage no priority is given to the attributes or parameters shown in the table.*

## 2.2 State of the art of microsystems for wine processing at commercial and at R&D levels

In this section the scientific progress beyond the state of the art of the different solutions will be presented. However, first the current methodologies used in winemaking control are summarised in order to better delineate the real scientific and commercial benefits of the miniaturised systems to be developed.

### Main laboratory based systems for the wine sector

Lots of parameters can and should be checked in winemaking. The OIV, *International Organization of vine and wine*, suggests appropriate analytical methods for them. A set of those methods are covered also by a Commission Regulation (2676/90) at EC level. Table 2.2 shows some examples:

<i>Parameter</i>	<i>Method/technique</i>
pH	Determination by pH meter
Acetic acid	Distillation and titration with standard base / Enzymatic method
Ethanol	Density and refractive index or is a distillation followed by hydrometry / Enzymatic method
SO <sub>2</sub>	Ripper procedure or aeration-oxidation method/ Enzymatic method
Total Acidity	Potentiometer method or acid base titration
Metals ( Current and heavy metals)	AAS
Conductivity	Conductimetric method
Acetaldehyde	Enzymatic method
Tartaric, Malic, citric and lactic acid	Enzymatic method or HPLC
Methanol, n-butanol, isobutanol, and isoamyl alcohol	GC-FID
4-ethylguaiacol	GC-FID, HPLC
H <sub>2</sub> S	GC-FPD
Residual Sugars	Rebelein method
Assimilable nitrogen	Sorensen method
Pesticides	HPLC, AAS, GC-ECD
Ochratoxin A	HPLC
<b>Water</b> / NO <sub>3</sub> <sup>-</sup> and NH <sub>4</sub> <sup>+</sup>	Spectrophotometry
<b>Water</b> / Matter in suspension	Gravimetry
<b>Water</b> / Electrochemical oxygen demand	Titrimetric method or spectrophotometry
<b>Water</b> / Biological oxygen demand	5-Day BOD Test or Spectrophotometry

GC-FID: Gas Chromatography- Flame Ionization Detector  
 GC- FPD: Gas Chromatography - Flame Photometric Detector,  
 GC-ECD: Gas Chromatography- Electron-Capture Detector  
 HPLC: High Performance Liquid chromatography  
 AAS: Atomic Absorption Spectroscopy

*Table 2.2: Summary of some techniques traditionally used in the winemaking sector*  
 Covering the above issues requires different laboratory-grade equipments such as Gas and Liquid Chromatography, Atomic Absorption Spectrometry, Spectrophotometers, together with some enzymatic methods, and some old-school wet chemistry methods.

Large wineries can afford laboratory equipment (each of them costing several tens of thousands of Euros). They can also employ full-time oenologists or technicians and “keep their skills sharp, and their reagents fresh”, by providing them with enough samples requiring routine analysis each week. On the other hand, small wineries, which are the common case, do not have such material and personnel resources, and they usually outsource to certified labs many of those analyses. This nevertheless implies a considerable cost. Annual bills of 30,000 € are not strange depending on the winery size and the type of wine. Here in table 2.3 follow typical cost per sample in one of those certified labs given external service.

Analysis	Cost price/u in a Laboratory
pH	2-10 €
Acetic acid	5-50 €
Total acidity	2-50 €
Acetaldehyde	13-80 €
Current metals: K, Mg, Ca, Na, Fe, Cu	13-80 €
Heavy metals: Pb, Zn, Cd, ...	18-80 €
Ethanol	4-40 €
4-ethylguaicol	13-80 €
SO <sub>2</sub>	5-50 €
H <sub>2</sub> S	5-50 €
COD	24-80 €
COD5	25-80 €
Conductivity	4-40 €
Matter in suspension	11-40 €
Nitrite	10-40 €
Assimilable nitrogen	4-40 €
Malic, lactic, citric acid	8-80 €
Residual sugar	5-50 €
Methanol, n-butanol, isobutanol, isoamyl alcohol	14-80 €

*Table 2.3: typical cost per sample of analysis done in traditional laboratories*

In addition to cost issues there are also time issues. Results from the certified laboratories can, if performed as expensive “quick service”, take two or three days. If done in-house, some of those analyses are long, tedious, cumbersome, prone to operator error, and on occasions difficult to interpret. Due to those issues, small wineries, which are the more numerous, don’t typically conduct enough routine analyses. In an ideal world such small wineries would be analysing hundreds of samples per week, while a large one will be facing tens of thousands.

Certainly, some laboratory automation has been progressing in this field. These auto-analysers make easier for the winery to perform these routine analyses, more quickly and with good analytical precision. Automated equipment may not replace skilled lab technicians because they still need to be maintained and calibrated, but it is true that they have a multiplication effect that enables one skilled tech to analyse more samples per day.

The automated equipment available tends to be specific for a given parameter. It is the case of SO<sub>2</sub> determination for the Hanna Instruments HI 84100<sup>11</sup>, the Mettler Toledo DL22<sup>12</sup> or the semi-automatic Vinmetrica SC-100<sup>13</sup>, for instance. Some enzymatic platforms enable a couple of different determinations using different kits onto the same low cost colorimeter (Megazyme L-Malic and Glucose/Fructose Test Kits<sup>14</sup>) and some hand-held digital refractometers have been made available (Misco Products Digital Wine Refractometers<sup>15</sup>) for sugar and alcohol determination.

Some analysers apt to multiparametric determinations are also available in the market, such as Lisa 200 Wine Analyser System<sup>16</sup> from Hycel Diagnostics. Alternative systems are Gibertini<sup>17</sup> semi-automatic Winematic, and fully automated multi-stage Wineflow. They are based on wet chemistry protocols with appropriate reagents and optical reading allowing the measurement of different acids, sugars, metals among other parameters. A special mention is deserved by rapid broad-spectrum auto-analysers, like the FOSS WineScan series, which is presented as an almost complete “lab in a box”. The FOSS<sup>18</sup> WineScan SO<sub>2</sub> is able to analyse free and total SO<sub>2</sub> alongside up to the 32 other parameters of interest. It is a complete (gas and liquid) FTIR system improved from the automation point of view. It is a fast but bulky and expensive machine that indirectly measures tens of parameters from infrared spectra relying in mathematical modelling and an extensive calibration database. Because of its broad-spectrum, physical and chemical approach (infrared), fastness and minimum use of reagent, it could be considered as an appropriate benchmark for the proposed developments.

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<sup>11</sup> <http://www.hannainst.com/>

<sup>12</sup> <http://us.mt.com/us/>

<sup>13</sup> <http://www.vinmetrica.com/>

<sup>14</sup> [http://www.megazyme.com/wine\\_brochure.pdf](http://www.megazyme.com/wine_brochure.pdf)

<sup>15</sup> <http://www.misco.com/>

<sup>16</sup> <http://www.clubafaceri.ro/21764/analizor--pentru-biochimie-lisa-200-plus-47269.html>

<sup>17</sup> <http://www.gibertini.com/>

<sup>18</sup> <http://www.foss.dk/industry-solution/products/winescan-so2>

Other companies developing new analytical instruments that have launched specific automatic analysers for the winemakers are Biosystems<sup>19</sup> (with photometric reading) and Biolan<sup>20</sup> (with multiparametric biosensors). Not only for the fermentation but for the whole process. Bruker<sup>21</sup> has also a Pesticide Screener system based on mass spectrometry for wine and Inbea<sup>22</sup> have developed also biosensor kits adapted to the wine monitoring. There are other small companies that have started developing systems based on MEMS, microsystems, and microfluidics technologies but the market covered is not yet important. Interesting examples are Jobst Technologies<sup>23</sup>, and Romelgen<sup>24</sup> with temperature sensors, MEMS, biosensors and microfluidics solutions that can be used in the beverage sector. Technobiochip<sup>25</sup> has developed biosensors, DNA chips and e-noses for wine monitoring. Opalux Inc<sup>26</sup> is also producing a photonic crystal based e-nose for bacteria detection based on the analysis of the by-products of the bacteria growth and useful for sensing heavy metals in water. Alpha-MOS<sup>27</sup>, one of the most well-known e-nose and e-tongue systems manufacturer for many industrial applications, also has instruments based on microfabricated sensors for chemical and organoleptic testing of soft and alcoholic drinks. In another direction, Aquamarijn<sup>28</sup> has also developed MEMS microsieves that could be interesting for the beverage sector, too.

Finally, a set of companies have started to commercialise RFIDs and tags that include sensors (basically temperature and humidity sensors) and that can be useful for the logistics of beverages. Among others we mention Kelsius Systems<sup>29</sup> and the big players KSW<sup>30</sup>, KBS<sup>31</sup> and Infineon<sup>32</sup>. In fact, RFIDs with sensors are today the only success case for Microsystems products in the food and beverage.

A similar situation can be found for water analysis, Agilent<sup>33</sup> and Varian<sup>34</sup> high performance cost systems are used in the traditional labs, and only a few simpler systems for single measurement parameters in water are available in a portable format, but no complete system is available. Neosens<sup>35</sup> is developing a new family of products using MEMS chemical sensors for controlling water contamination.

Only few of the above alternatives are really handheld devices: most of them are bench-top systems and none of them have been developed for continuous process

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<sup>19</sup> [www.biosystems.com](http://www.biosystems.com)

<sup>20</sup> [www.biolanmb.com](http://www.biolanmb.com)

<sup>21</sup> [www.bruker.com](http://www.bruker.com)

<sup>22</sup> [www.inbea.com](http://www.inbea.com)

<sup>23</sup> [www.jobst-technologies.com](http://www.jobst-technologies.com)

<sup>24</sup> [www.romelgen.ro](http://www.romelgen.ro)

<sup>25</sup> [www.technobiochip.com](http://www.technobiochip.com)

<sup>26</sup> [opalux.com](http://opalux.com)

<sup>27</sup> [www.alpha-mos.com](http://www.alpha-mos.com)

<sup>28</sup> [www.aquamarijn.nl](http://www.aquamarijn.nl)

<sup>29</sup> [www.kelsius.com](http://www.kelsius.com)

<sup>30</sup> [www.smartrac-group.com](http://www.smartrac-group.com)

<sup>31</sup> [www.kbs.fr](http://www.kbs.fr)

<sup>32</sup> [www.infineon.com](http://www.infineon.com)

<sup>33</sup> [www.chem.agilent.com](http://www.chem.agilent.com)

<sup>34</sup> [www.varianinc.com](http://www.varianinc.com)

<sup>35</sup> [www.neosens.com](http://www.neosens.com)

and none address yet the wine sector application with a complete set of new sensing instruments based on MNT and Smart Systems. However, it is well accepted by industry that microsystems integration can bring further size and cost reductions, definitely contributing to the desirable ultimate in-line/on-line monitoring target. It could also help to build bench-top equipment similar to the described above, smaller, affordable and multi-parametric in purpose.

### ***Comparative cost of wine oriented instruments***

On a small scale production scenario the cost of the microsystems involved in any of the prototypes of this proposal will be in the range of *several tens of Euros to few hundred of Euros*, so selling prices could be in the range of a few thousand Euros (1-12 k€). The simpler systems such as the gas sensor system and the water quality system will be in lower range, while the MIR system and the micro-GC/LC systems, more demanding from an integration point of view, will be in the medium and higher range, respectively. Microsystems share with microelectronics the economy of scale concept, bigger production meaning lower cost per unit, so the above range could be regarded as an upper limit.

‘Selling price’ is, of course, a market determined parameter. As a background, the figures mentioned above are at least one or two order of magnitude lower than usual multiparametric lab equipment (GC, HPLC, AAS – 50-100 k€). It is not with those systems that our developments will have to compete, but this price difference determines having or not having on-site process control for small and medium wineries. The proposed systems will indeed positively compare with more affordable options such as colorimetric/enzymatic automatic wine analysers (20-40 k€), semi-automatic and automatic titrators (7-15 k€), and they are in the range of enzymatic biosensors (1-4 k€). The different of price in each segment will depend on brand issues, complexity of the systems, and the degree of automation of the measurements: in the simple ones, sample must be prepared by the user and feed into the machine. In addition, all these equipment need long-term unstable and expensive reagents for their operation contributing significantly to their cost of ownership (depending on how intensive the use, it could represent each year a significant fraction of the most expensive examples or amount to more than the cost of the less expensive sensing systems themselves). This is also the case for the cheapest systems available (0.3-0.6 k€), such as portable refractometers or devices intended only for approximate SO<sub>2</sub> levels determination.

On the other hand, reagent-free FOSS products are situated in the 30-150 k€ range (OenoFoss and WineScan SO<sub>2</sub> being the two extreme products), which even in the lower range is a price still too high for small and medium wineries. WineScanSO<sub>2</sub> is only advertised for large wineries and certified labs.

From the described situation it is clear that there is a real need in for carrying out R&D on MST and Smart Systems for developing cheap, portable, fast and reliable

systems for the wine sector in particular, but extendable to the beverage and food sector in general. The systems would cover a range of applications of medium to low cost, high portability, automation and low or zero reagents consumption, as shown in figure 2.4. This is a good way, on the one side, to improve the analytical capabilities of the SME of the wine sector that cannot afford the cost of complete laboratories and very expensive instruments; and, on the other side, to work steadily towards in-line/on-line detector systems which are of interest for wine producers regardless the company size.

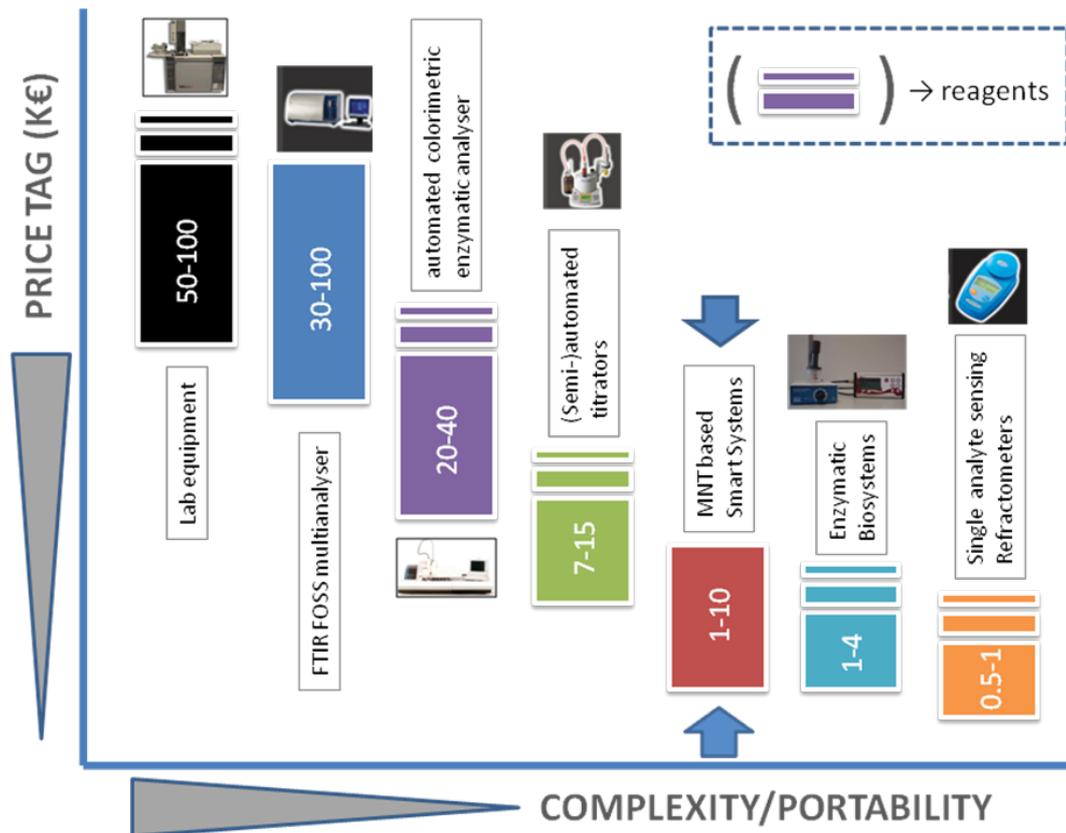


Figure 2.4: Cost of the proposed MST based systems compared to other wine oriented instruments

## R&D on new analysis tools

In the last years an interesting R & D activity has been carried-out in the field of development of new materials, devices and systems that can be useful for the beverage industry. Within the FoodMicroSystems project a work of identification of key publications for the entire food sector was done and in this section we analyze the results of this literature search for the beverage industry, primarily for wine and beer. These beverages have a complex elaboration process, including the step of fermentation which is vital to the final product quality. The following table is a summary of the main results published mainly on the development of new sensors and systems based on microtechnologies for improving their performances or for looking at new applications that were solved only with laboratory systems until now.

It can be seen that there are different types of families of sensors that show good potentials for various applications in the field of beverages. We can highlight the possibilities of various types of mechanical, chemical and biochemical sensors, although the performance of these last ones in terms of stability, repeatability, ease of use, response time and cost have not yet come to be sufficient to reach the market. Furthermore, because of the complexity of some of the parameters involved in the beverage quality control there is a very important activity in developing not only specific singles devices but systems based on arrays of several sets of non-specific sensors that together with specific hardware and software for sample pre-processing and data analysis, may improve the performance of the complete systems. Among them, electronic noses and electronic tongues are the most popular in terms of R & D, and various applications for qualitative and quantitative evaluation have been tested. However, the lack of specificity and the need for the improvement of all its components make them not yet fully accepted in real applications.

Finally, an additional improvement would be the combination of olfaction, taste and vision for improving the overall specifications of the system in order to mimic sensory evaluation and tasting panels, or even to combine them with micro implementations of typical laboratory systems, such as mass chromatographs.

From table 2.4 it can be also inferred that the main objective of the R & D studies in the beverage sector has been the determination of the final product quality for discrimination, sorting, authentication, etc..., but not so much the process development and control. The main targets addressed up to now have been the determination of the attributes and general organoleptic parameters of wine, beers or soft drinks determined at vapour and liquid phases, using devices that increasingly rely more on the microtechnologies. In conclusion, there are multiple beverage applications that from the point of view of MST technologies, devices and systems, are attractive and that must be discussed together with the view of beverage producers to see which families of sensors and systems are the most attractive and offer more possibilities to real solve problems. This will be presented in the following sections.

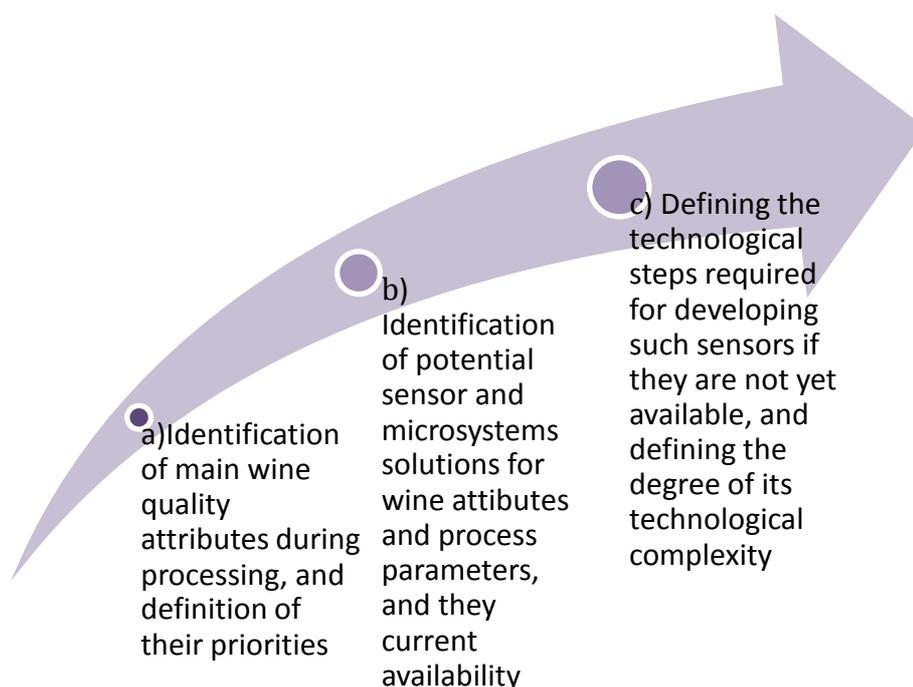
Device / System	Application	Reference
<b>Gas sensors + arrays</b>	quality monitoring	[Adami-2006]
	alcoholic fermentation	[Maciejewska-2006]
	classification of wines and beers	[Ghasemi-2012], [Lozano-2009], [Villanueva -2008]
	discrimination	[Wongchoosuk-2009], [Lozano-2007a], [Santos-2005]
	yeast cells growth	[Ress-2009]
<b>Electro-chemical sensors, ION, ISFETs</b>	pH, ions	[Artigas-2003]
	quality monitoring	[Pigani-2011]
	classification	[Gutierrez-2011]
	Safety: OTA	[Perrotta-2012]
	waste water contamination	[Orozco-2008]
<b>Biosensors &amp; arrays</b>	alcoholic fermentation	[Monosik-2012], [Piermarini-2011], [Goriushkina-2009], [Wang-2004]
	antifraud	[Bucur-2008]
	glucose in wine	[Tian-2007]
	glucose in grape	[Alonso-2005], [Payen-2005]
	vitamins in juice	[Hauhey-2012]
	sugars in sodas and coke drinks	[Szollosi-2011]
	profiling whisky	[Yoshida-2012]
	soft drinks process control	[Murakami-2010], [Zhang-2007]
	green tea classification	[Fu-2012]
	fungi contamination in coffee	[Sberveglieri-2011]
	quality of bottled water	[Men-2011]
	safety: tyramine, SO <sub>2</sub> , bacteria	[Lata-2011], [Martinez-2011], [Pires-2011]
	antioxidant capacity, polyphenols	[Photikin-2010], [Elkaoutit-2008], [Campaanella-2004], [Gomes-2004]
<b>Colour sensors</b>	wine quality	[Torre-2009]
<b>Conductive sensors</b>	alcoholic fermentation	[Colombie-2008]
<b>MEMS resonators</b>	rheology, sugar, alcohol contents	[Paxman-2012], [Fu-2008]
<b>MEMS spectrometers</b>	process control of carbonated beverage	[Kenda-2010]
<b>Temperature sensors</b>	intelligent packaging	[Brody-2010]

<b>Capillary electrophoresis separation modules</b>	safety	[Benvenuto-2008], [Fu-2008]
<b>Optical sensors</b>	safety, SO <sub>2</sub>	[Silva-2006]
	phenolic maturity	[Zerovic-2008]
	alcoholic fermentation	[Jimenez-2011]
<b>Ultrasound sensors</b>	sugar, ethanol	[Schock-2010]
	fermentation	[Watson-2010]
<b>e-nose</b>	aromatic compounds of product and raw materials	[Ceto-2011], [Ghasemi-2011], [Rudnitskaya-2010], [Vera-2010], [Ragazzo-2009], [Torre-2009], [Chang-2008], [Lozano-2006], [Santos-2004]
	authentication, classification, discrimination	[Cabañes-2009], [Garcia-2006], [Schafer-2006], [Pinheiro-2005], [Lozano-2005], [Sayago-2003]
	safety: pathogens, brettanomyces	[Nermelstein-2008], [Cynkar-2006]
	fruit maturity & decay	[Athamneh-2008], [Zarzo-2005]
	ageing & evolution	[Prieto-2012], [Lozano-2008], [Lozano-2007b]
<b>e-tongue</b>	adulteration	[Stoj-2011], [Ghasemi-2008], [Parra-2006]
	process improvement	[Ghasemi-2012b], [Gay-2010], [Moreno-2008]
	taste attributes	[Zhou-2010], [Rudnitskaya-2009], [Arrieta-2010b], [Ghasemi-2009],
	classification, discrimination	[Ghasemi-2012a], [Arrieta-2010a], [Gutierrez-2010a], [Kantor-2007], [Tian-2007], [Parra-2004], [Srichaisiriwech-2010], [Polshin-2020]
	quality control, tannins, phenols	[Gutierrez-2010b], [Labrador-2009], [Zeravik-2009], [Puech-2007], [Lvova-2006]
	quality of bottled water	[Men-2011]
	safety, sulphites, defects	[Mednova-2009], [Ceto-2012], [Francioso-2007]
<b>Coupled e-nose and e-tongue</b>	impurities in water	[Ferreira-2007], [Vlasov-2005]
<b>electronic panel: nose, tongue, eye</b>	Organoleptic characteristics	[Casale-2010], [Swiegers-2005], [Buratti-2004]
	effects of type of closure	[Prieto-2011], [Rodriguez-2004]
<b>Coupled gas chromatographer and e-nose</b>	classification and discrimination	[Garcia-2011], [Ragazzo-2009], [Beltran-2008], [Ragazzo-2008], [Ragazzo-2005]

Table 2.4: Summary of R&D works carried out on sensing devices and systems for the beverage sector mostly devoted to MST technologies.

### 3 Roadmap for process & quality control of wine and beer

In this chapter, the results of the different meeting held specific with the wine and beer sector are presented. Discussions started with the results shown in the former chapters and now the objectives of the meetings with specialists were to develop “wine application driven technology roadmaps” for the microsystems sector that can draw the foundations for new analytical systems. The roadmap process is being carried out in different steps being the main ones as shown in figure 3.1.



*Figure 3.1: Main steps followed for the roadmap process*

The preliminary information for defining the roadmaps has been obtained in open discussion events specially organised by the FoodMicroSystems project among wine producers, oenologists, analytical instrumentation companies and Micro and Nano Technologies developers and researchers mainly from Mediterranean countries. The results of the discussions of points a) and b) give as result a set of diagrams (for example, see figure 3.2) that graphically show the priority in time (three, five and ten years from now) of each parameter according to the winemakers point of view and the associated sensor and/or system solution and its degree of complexity, according to the experience of MST and instrumentation developers participating in the open discussions. Point c) is elaborated later in Deliverable 4.5 for the more challenging and critical set of systems identified.

In the next paragraphs the graphical outcomes of the FoodMicroSystems study of the main wine process steps and check points are presented and discussed.

### 3.1 Roadmap for quality, safety and authentication of raw materials at the reception point

#### Opportunities



The main objective is to develop new on-line sensing systems usable at the weighbridge and the reception point in order to accept and qualify the incoming material. Not all wine producers are interested in investing on systems for the determination of the quality of grapes at this stage as in some cases, they also cultivate their own grapes under their control and thus, their quality is monitored and evaluated at early stages. However, some other elaborators buy most or your wine and not grapes, and thus their interests also differ.

In the case of the analysis of the grapes coming from the vine, it is of high importance to develop new systems (if possible, to be multiparametric) that show clear advantages in terms of cheaper and faster response (maximum 5 to 10 minutes response) compared to the traditional analysis in the laboratory, as this means that a larger amount of grape can be evaluated and accepted for its elaboration without delays, preventing from any safety issue.

However, when dealing with raw materials, it is not only the determination of the quality of grapes or most but also of wood barrels, glass bottles, corks, yeasts and any other material that interacts with the wine during its life. For the acceptance of these raw materials, the interest is building systems that allow increasing the amount of material being analysed in an as short period of time as possible in order to increase productivity.

In the case of beer, malt is a dry material that does not perish so fast and accepts longer analysing periods, despite, fast response is also a major interest for increasing productivity. Humidity and sanitary aspects of cereals is of interest for elaborators. The use of metallic cans and barrels for packaging of beer is another difference compared to wine.

Finally, for soft drinks and sodas, no other issues are addressed apart from the control of the specific raw materials involved in each case.

#### Needs

The main interests in the wine sector for the grape acceptance are related to their quality and (i.e. aromatic richness, phenolic maturity, gluconic acid, colour ...) and sanitary status (presence of microorganisms). Screening good quality grapes and at

the right ripeness degree is vital for achieving high production yield during wine processing.

Of course, it is also important to know the toasting level, cleanness degree and sanitary status of wood barrels (*brettanomyces...*), corks (TCA), and glass bottles. Specific for the beer is the quality and safety control of malt and other grains and sanitary status of metallic cans and barrels.

Authentication of origin of raw materials may be also an issue for some specific cases.

## Roadmap

Figure 3.2 shows the graphical result of the roadmapping discussions done together with wine and brewers and MST and instrumentation experts. In orange boxes the main attributes and parameters of interest for the sector are laid-out. X axes gives the priority for a new solution for the identified parameters. The possible kind of microsystems technology or device that may contribute to improve analysis methodologies are laid-out as blue arrows associated to the corresponding orange box. The complexity of the MST solution according to the experts know-how is shown relating its position with the Y axes. The identification of a potential MST solution may be because it has been concluded that an existing device can be improved with MST or that a development of a new technology or MST that accomplishes the required specifications seems feasible. For long-term objectives it could be also possible that this feasibility cannot be already assessed, but that the experts have considered that it is worth and challenging to develop new systems for those applications.

From the prioritisation of winemakers, it is seen that quality of grapes is of most importance and novel and more performing systems should be desirable at short term. Quality and screening of other materials are left as medium to long term priority.

From the MST complexity and availability of solutions it is observed, that parameters related to physical measurements are considered of low complexity and with chances of developing novel solutions at short term, while solutions based on bio-sensing are more medium to long term because of lack repetitiveness and sampling complexity associated to current state of the art.

Finally, cork and wood quality is another challenging application for microsystems based sensors and systems. It is also agreed that artificial vision systems (based or not on microsystems) can be of use for some analysis if fast and smart enough. However, screening solutions like the systems based on recognizing green grapes from those who are not and eliminate those that are not mature still has to be optimised and increase in specificity. Probably, the combination with microsystems would help on improving the criteria of selection.

Figure 3.2 also shows that already existing solutions based on e-nose and e-tongue are not yet satisfactory for the elaborators and need for further research for improving their performances and being much more specific according to the applications requirements. These new solutions, despite the interest are considered of high complexity from the MST experts and it is not clear that they can match the requirements at short term.

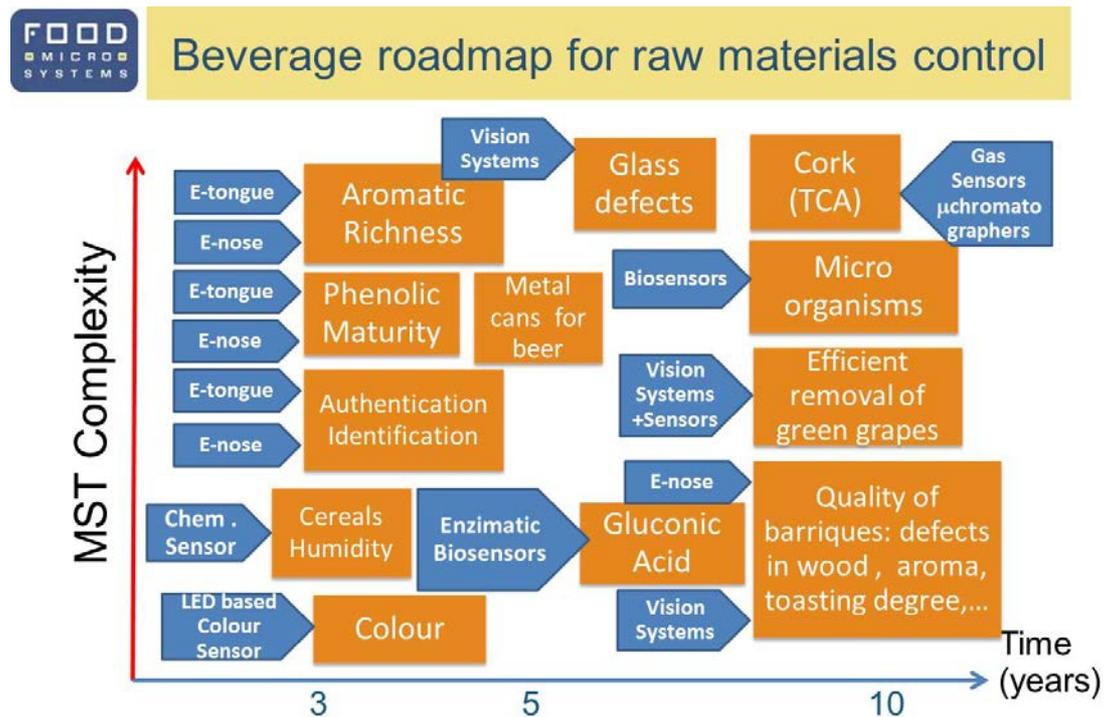


Figure 3.2. Roadmap of main quality and authentication indicators for raw materials

### 3.1 Roadmap for the fermentation process in tank

#### Opportunities



Fermentation is the most important step of the wine elaboration process. Thus, it is important to control it as much as possible. It is difficult to speak generically about the fermentation process as there are noticeable differences among the elaboration of different types of wines (i.e. between red, white and rosé wines, or between still and sparkling wines, or sweet wines, etc...) and beers (dark, light, alcoholic contents,...). However, some generic needs can be found for all products. Fermentation may occur in one or more steps (fermentation, stabilisation, second fermentation) and media (tank, barrel and bottle) and this gives again more degrees of differentiation among wine and beer processes. Another

opportunity is the interest for many elaborators for growing by themselves and controlling the yeasts to be used during the whole process. Developing systems that can be attached to each tank and give continuous information would be a real advantage compared to hand made current sampling and analysing processes.

## Needs

Improvements are particularly important nowadays for the control of the fermentation step, which, despite being the cornerstone of the wine production process, is still made off-line. A real improvement could be to have a continuous (one measurement every hour) multiparameter on-line control instead of one measurement per day in the laboratory.

The main parameters of interest identified during the roadmapping discussions of FoodMicroSystems project were: density, sugar contents, colour, temperature, conductivity, viable yeast counting and viability, acidity, presence of mercaptanes, lactic bacteria and polyphenols (types of polyphenols vary from one wine to another and it is more interesting to make a profile of the different families of polyphenols in the wine), Sulphurous Anhydride, Nitrogen and Oxygen contents. The concentration of major physical and chemical parameters of a fermenting most but also other chemical compounds is usually provided by analytical laboratories, often 48h-1week after sample preparation, and costing up to 2,000-30,000 euros yearly depending on the winery size.

Viable yeasts are important to conserve. Devices that measure not only the viability of microorganisms but also the vitality and vibrancy could be interesting during the fermentation.

Some Special parameters for beer industry are the assessment of the elimination of DMS (Dimethylsulfide) and its precursors before the fermentation. Now, the measurement is made by HPLC or GC and is very long (one day). This step is expensive and must be shortened. The bittering hops are boiled in the wort for around an hour to an hour and a half. This long boil extracts resins, which provides the bittering. There is a need of a portable device able to measure in a boiling liquid, which is very difficult.

During the ripening, diacetyl/vicinal diketone (VDK) is an important parameter to control. It is measured by Gas Chromatography (2-3hours). Thus, it could be interesting to have a device to easily and fast measure these parameters (DMS and VDK) to save time, money and energy during processing.

Monitoring bitterness during filtration is much more important than in the tank because of normal losses at this step. For brewers losing bitterness means losing money.

An indicative specifications list about lower and upper limits proposed for substances of interest is reported in table 3.1 obtained from oenology reference works. In the same table a separated column reports the phase of the detection methods that could be approached with microsystems. Table 3.2 also refers to key parameters to be measured during fermentation.

Compound	Lower limit	Upper limit	Detected phase
<b>pH</b>	2.5	4.5	Liquid
<b>Assimilable Nitrogen</b>	0.05 g/L	1 g/L	Liquid
<b>Acetic acid</b>	0.1 g/l	1.5 g/l	Liquid
<b>Acetaldehyde</b>	0.05 g/l	0.5 g/l	Liquid
<b>Magnesium</b>	50 mg/l	200 mg/l	Liquid
<b>Ethanol</b>	50 g/l	200g/l	Gas/Liquid
<b>4-Ethylguaiaicol</b>	50µg/l	1.5 mg/l	Gas
<b>SO<sub>2</sub></b>	50 mg/l	250 mg/l	Gas
<b>H<sub>2</sub>S</b>	0.1 µg/l	1.0 µg/l	Gas

*Table 3.1 : Summary of the main parameters used by oenologists*

Parameter	Range	Comments
<b>Alcohol content</b>	0-15% vol	low values in the must, high values in the wine
<b>Total Acidity</b>	3-9 g/l	similar values in must and wine
<b>Volatile Acidity</b>	0-1 g/l	low values in the must, high values in the wine
<b>Malic acid</b>	0.1-3 g/l	high values in the must, low values in the wine
<b>Lactic acid</b>	0-3 g/l	low values in the must, high values in the wine
<b>Sugars</b>	2-200 g/l	high values in the must, low values in the wine

*Table 3.2: Summary of ranges of interest of other attributes of wine*

Other indicators of fermentation can come from the measurement of volatiles. Examples of volatiles produced during fermentation including their normal concentration range are shown in table 3.3.

Compound	Normal range during fermentation
<b>Methanol</b>	0 - 200 mg/l
<b>Ethyl acetate</b>	0 – 100 mg/l
<b>Butanol</b>	0 – 8.5 mg /l
<b>Acetaldehyde</b>	0 - 0.05 g/l
<b>Propanol</b>	0 -70 mg/l
<b>Ethanol</b>	0 - 100 g/l
<b>3-methyl-1-butanol</b>	0 – 500 mg/l
Source: Wine Science by R.S. Jackson, Academic Press, 3rd revised edition, 2008, ISBN-10: 0123736463	

*Table 3.3 : Other Indicators specific for fermentation*

## Roadmap

In fermentation priority is given to the automatic control of the processing parameters of this important step. The rheological parameters that give information of the sugar contents and degree of alcohol are also of capital importance at short term for elaborators together with quality attributes. At medium term there is the need of developing better systems for Ion, vapours and gases dissolved, that are also related to the evolution of wine during fermentation. Finally, separation of sugars is also of interest but at longer term, as it is not vital for the process.

From the microsystems side, it can be observed in figure 3.3 that the improvements should come from the continuous on-line control of the kinetics of the fermentation process and other important quality markers. As for other processing steps, some solutions of lower complexity may be designed based on simple physical and chemical parameters but, complexity increases when solutions have to deal with new types of biosensors (especially genosensors).

Other parameters of interest can be only addressed with artificial vision systems, but they are not considered microsystems.

## Roadmap for process control in the tank

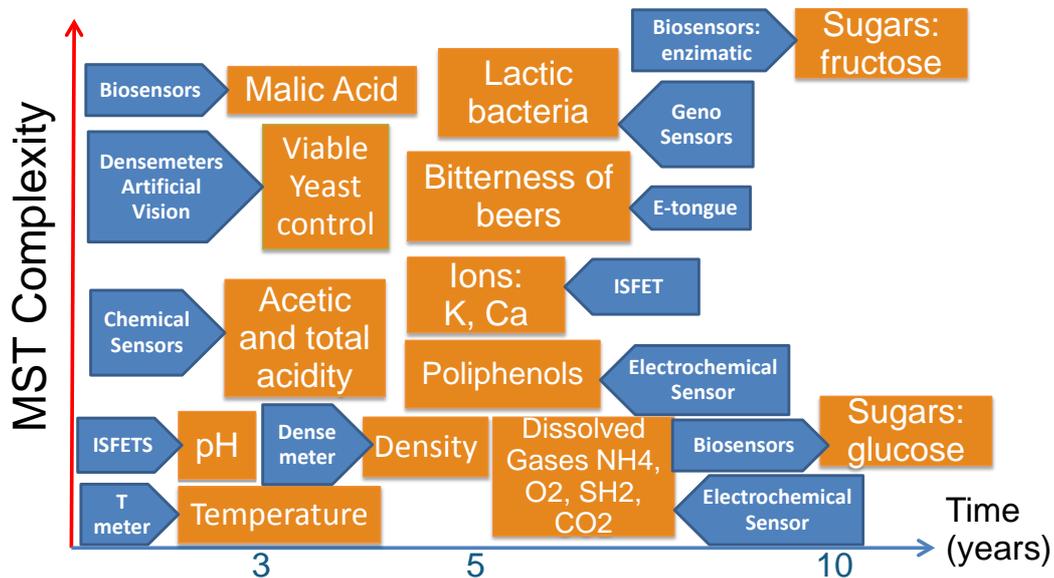


Fig. 3.3. Roadmap of main quality indicators of wine fermentation process

## 3.2 Roadmap for the control of ageing in barrel

### Opportunities



For mature and sparkling wines, after the fermentation and stabilisation steps in a tank a period of maturation and ageing is usually performed in wood barrels. This step is also usual for some kinds of beers. The objective when developing new systems usable in this processing step is again to develop portable on-line multisensing systems for keeping track of the attributes and parameters of the wine or beer being processed.

### Needs

Needs in the barrel are similar for the fermentation processing and in addition, here, the main parameters of interest are the colour and pH, the Oxygen contents (oxygen consumed in barrel/tank during the wine making to follow it through the time), the monitoring of the tartaric acid, the free SO<sub>2</sub>, the acetic acid, the redox potential, the phenols and biogenic amines presence and of other alternative microorganisms like *brettanomyces*. Ageing may last long periods of time, and thus there is a need of continuous monitoring of key parameters.

## Roadmap

Figure 3.4 shows the layout of priorities for the quality indicators and the complexity of the MST solutions. There is the need of monitoring the evolution of such indicators over time, but in this case it is not expected that a cost efficient solution may be developed for attaching one multisensing system to each barrel of a process plant. Thus, in this case, the objective is to develop a portable system that can be used in-line for testing barrels in a sequential mode.

For crucial analysis (i.e. tartaric acids, free SO<sub>2</sub>...) it is necessary to develop complex chemical and bio-chemical sensors, as for the detection of microorganisms that can come from the process or from the barrel itself. These systems are seen as of high complexity and available at mid to long-term. On the other hand, parameters of high priority that can be addressed with already known physical and simple electrochemical sensors and devices may be developed faster.

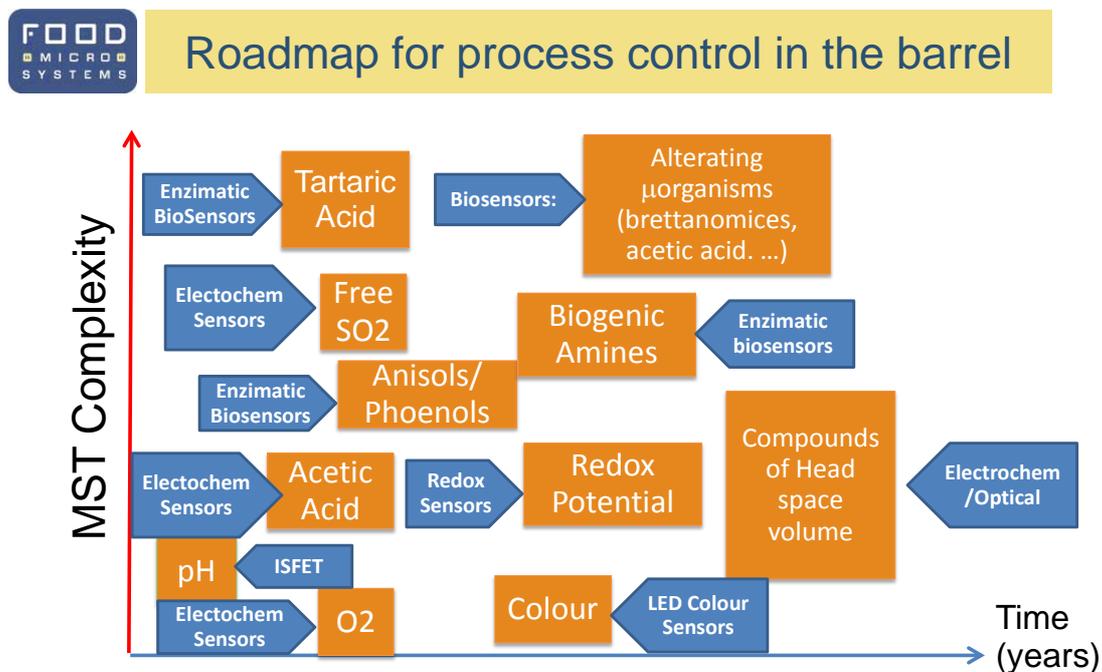


Figure 3.4. Roadmap of main quality indicators during ageing in a barrel

### 3.3 Roadmap for process control during bottling

#### Opportunities



Bottling is another step in which many different processes are involved. Bottling can be the final process of packaging of the product already elaborated, but in some cases (i.e. champagne and cava processing), it is an intermediate step

of the process, as the product is still evolving within the bottle, and second fermentation processes can occur in them. For the first case, the opportunities for novel microsystems based systems are related more to the quality and safety aspects of the final product, while in the second case, the analysis needs are more related to the continuation of the monitoring of the evolution of the product during this new phase in bottle, with similar indicators as for the evolution in barrels.

Prior of bottling, there may be some steps like the addition of sugars of liquors (tirage process in champagne and cava), or gases for preventing oxidation that are specific at this bottling stage. In all these steps, new improvements using novel micro sensors and microsystems would be of high interest.

Other typical aspects related to bottling are to improve and develop the sensing and control capabilities of the machinery specifically devoted to the bottling process. There are already existing solutions embedded within bottling machines used for controlling the level of the product after bottling, detecting defects in glass bottles and the presence of undesired particles. Inspection of bottles for ensuring that they do not content undesired particles is now being done with artificial vision, but could benefit from any kind of microsystem development that would help on increasing speed and productivity.

Developing multisensing systems that could be embedded into the bottle or event into the cork (the intelligent bottle or the intelligent cork visions) at very low cost is seen as a futuristic wish that would help very much in monitoring the evolution of the product with the bottle. Today, parameters that cannot be measured for the product in the bottle must rely on the results obtained during the processing in the former steps in the barrel or tank.

Similar requirements are met in the beer sector, but with the specificities related to the packaging with metallic cans and barrels, in addition to glass bottles. These systems make the determination of parameters from the outside of the packaging much more challenging.

## Needs

Main parameters associated to quality and safety of wine at this stage are related to its aroma (volatiles), taste (acids, phenols, pH...) colour and safety markers (amines, allergens, organisms,...) and thus, it is necessary to make measurements not only in the volume of the liquid but also in the headspace of the bottle, which is the space between the top of the liquid the bottom of the cork. For preventing the undesired evolution of the product after final bottling, oxygen contents should be minimised (and one control is to measure it). Optical techniques exist that allow measuring O<sub>2</sub> in bottle when it is closed looking at the Oxygen contents and CO<sub>2</sub> in the product and in the headspace, but systems are not cheap and simple. This is not an easy need that will require long term research on different microtechnologies.

Other parameters of interest in bottling that have to be monitored are: pH, sugar contents, precise percentage of alcohol, SO<sub>2</sub> and CO<sub>2</sub>, acidity limit, and also other indicators like colour and turbidity, taking into account that in many cases, glass bottles are coloured.

## Roadmap

Higher priority is given to systems that can help on improving the determination of quality parameters of the product, while defining as mid to long term need the replacement or improvement of the optical and artificial vision systems that are already established and used in the production lines. Some systems based on electrochemical are expected to be easily implementable in the near future, while others based on bio-sensing are expected to be long term as many of them do not yet exist up to now adapted to the application, as shown in the graphical layout of requirements and potential solutions of figure 3.5.

Specific for the beer is the filtration process, that could benefit from MEMS micromachined silicon filters with very well defined pores (100 nm and up), as already developed for dairy.

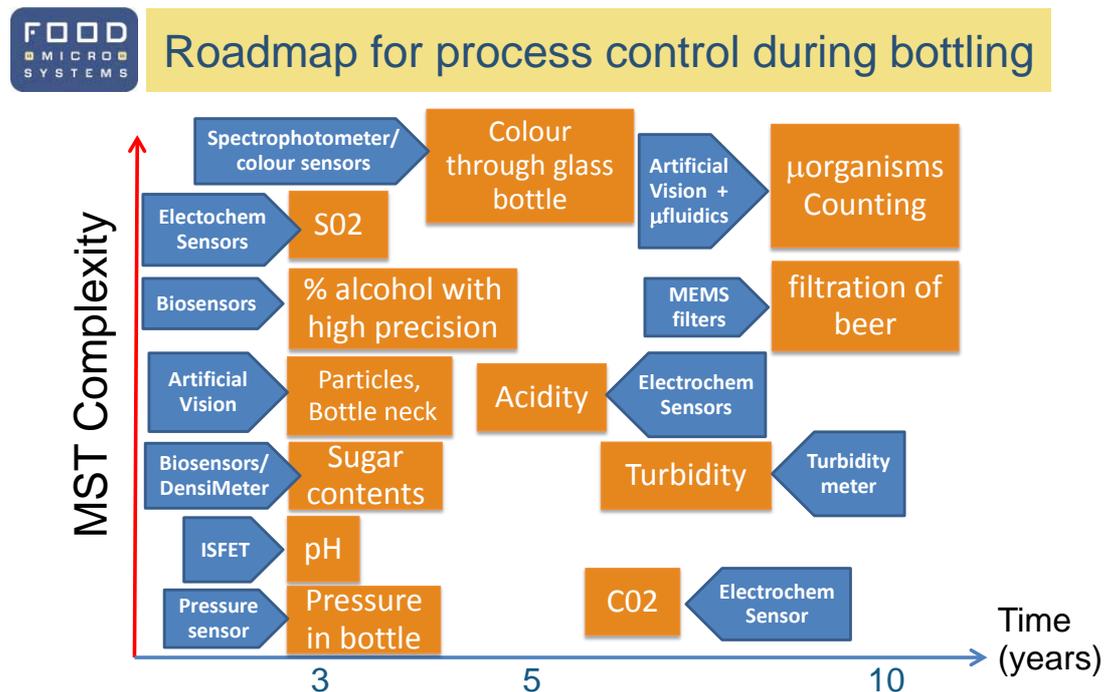


Figure 3.5: Roadmap of main quality indicators during bottling

## 3.4 Roadmap for logistics and authentication

### Opportunities



The quality and safety of wine and beer can be highly affected during the storage and delivery time to the consumer, if bottles or cans are exposed to extreme environmental conditions during storage inside and outside the plant and transport or shipment. As already stated wine and beer are products that are exported in big percentages and it means that logistics issues are of capital importance.

The incorporation of sensing capabilities in RFIDs specific for the product is very promising for the winemakers. The incorporation of sensors in the own label of the bottle is also a long term vision as far as very low cost can be achieved.

Moreover, in big beer plants, the localisation of lots of products with RFID would be a big help on the control of product.

### Needs

Systems that can monitor parameters like temperature, light and humidity are of high interest. Authentication of wine is another aspect that can benefit from specifically developed e-tongue and e-nose systems. The origin, variety of grapes, level of maturation of the product, etc... have to be controlled and classified if authentication or antifraud procedures have to be implemented. Parameters for this classification are related to the quality and safety attributes of the product mostly based on taste and aroma, as for the case of raw material characterisation.

### Roadmap

The integration of sensors in RFIDs but specific for the beverage sector seem to be a realistic implementation at short to medium term, and only the cost issue could prevent the success as far as technologies like printed electronics, for sensing development are not well established at the right cost.

E-nose and e-tongue solutions for classification are also potential solutions that should be accepted and desirable at short term, but there is still the need of optimising the sensors, separation devices, and the associated chemometry for signal processing. The implementation of intelligent labels with new types of sensors (not necessarily only based on micronanotechnologies but also on new sensing materials and/or inks) is a goal that has been defined as a longer term, as shown in figure 3.6.

## Roadmap for logistics and authentication

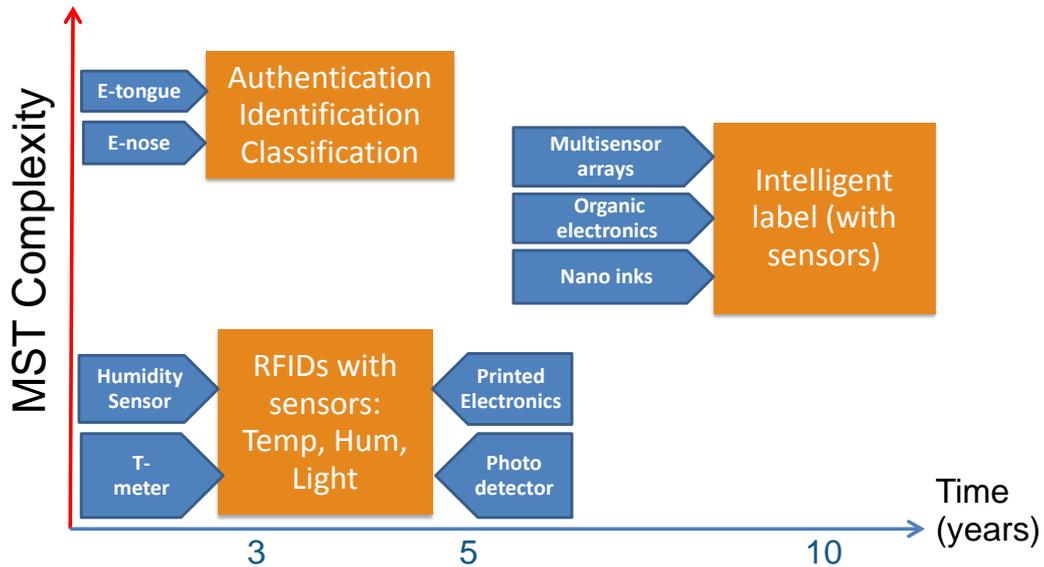


Figure 3.6: Roadmap of main parameters for logistics and authentication

### 3.5 Roadmap for monitoring safety in beverages

#### Opportunities



Safety is a main concern for both wine and beer and any other beverage and food product. Risks can come from different sources, use of pesticides in vine and land, sanitary status of grapes, cereals, or fruits, contamination coming from other materials in contact with the product during processing, incorrect processing and cleaning procedures, incorrect storage procedures, effects of additives on consumer health, etc.... In many cases, novel biosensing based systems would be an added value if performances are improved, and simplicity of use is achieved at the right cost.

Detection of harmful particles in the product is another concern related to safety of the consumer.

#### Needs

Pesticides are still commonly used in vines and control in the final product is necessary. On the other hand, sulphite is an allergen commonly present in wine and has to be labelled if its value is higher than 10ppm. However, the main indicators for safety use to be chemical products or microorganisms that may have an impact not

only on the safety but also on the quality of the product. The main parameters and priorities: biogenic amines, OTA toxins, pesticides in grapes, allergens (histamines) and microorganisms.

Finally, and water is becoming an increasingly demanded resource in spite of its decreasing availability, evaluation of its quality will be covered in the next section, but analysis of water for safety reasons is another aspect to be taken into account.

## Roadmap

In figure 7, the result of the discussion on safety is presented. It is seen that most of the novel MST solutions are expected to be based on bio-sensors and, thus, the degrees of complexity are scored as medium to high and as mid to long term vision.

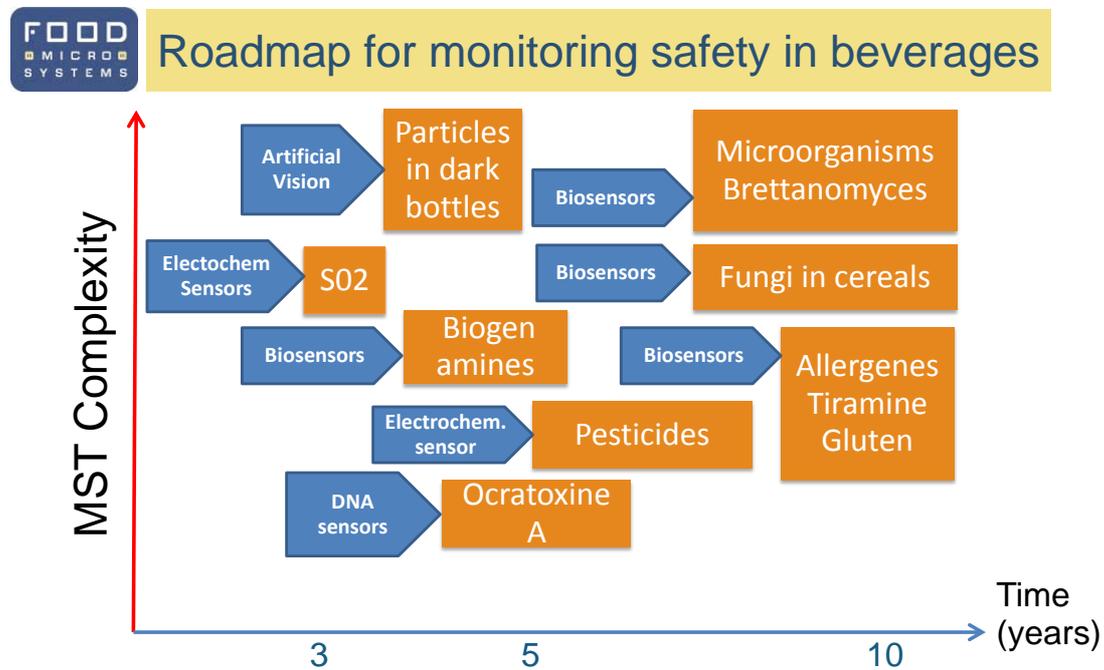


Figure 3.7: Roadmap of main parameters for monitoring safety

## 3.6 Roadmap for water quality monitoring in cellars and production plants

### Opportunities



As already mentioned water scarcity and cost may be an issue in the future for industrial activity in general and specifically for the food and beverage producers. Systems for saving water are crucial for the economic sustainability and many of them may be based on new microsystems.

There are also opportunities in the control of waste water and effluents that have to comply with existing regulations. Another aspect of potential interest is the need of measuring the amount of CO<sub>2</sub> that is lost in the environment for employee safety. Systems already exist for these applications but are not based on microsystems technologies. The real improvements achievable with new sensors and microsystems have to be evaluated especially if a high degree of integration level can be achieved, as this is probably the real advancement for the future.

## Needs

The contents of pesticides, pH, ions, the organic and chemical load in water indirectly measured through the determination of the COD (Chemical demand of Oxygen) and the BOD (Biological demand in Oxygen) and heavy metals are the most important needs for water analysis the beverage sector, as some of these parameters are used to determine the payment of environmental taxes if thresholds are exceeded. In tables 3.4 and 3.5, the main parameters defined in the present European directives related to water quality for consumption (Directive 80/778/EEC) and for water discharges in sensitive urban areas (Directive 91/271/EEC) are presented.

Parameter	Conc. ranges	LOD	Maximum admissible
pH	pH 2-13	pH 13	6.5-8.5 <sup>1</sup>
NH <sub>4</sub> <sup>+</sup>	5.6 10 <sup>-6</sup> - 2.1 10 <sup>-2</sup> M	1.8 x 10 <sup>-6</sup> M	50 mg/L <sup>1</sup>
NO <sub>3</sub> <sup>-</sup>	2.5 10 <sup>-5</sup> - 2.1 10 <sup>-2</sup> M	1.1 x 10 <sup>-5</sup> M	0.5 mg/L <sup>1</sup>
Conductivity	0.35 to 12.00 mS cm <sup>-1</sup>	0.12 mS cm <sup>-1</sup>	400 μS cm <sup>-1</sup> <sup>1</sup>
EOD	5-1400 mg/L O <sub>2</sub>	4.3 mg/L O <sub>2</sub>	125 mg/L O <sub>2</sub> <sup>2</sup>
Heavy metals (Cu)	0-10 μM	0.3 μM or 19.2 μg/L	2 mg/L <sup>2</sup>

1. Directive 80/778/EEC related to the quality of water intended for human consumption
2. Council Directive 91/271/EEC concerning urban waste-treatment

*Table 3.4. Summary of directives for water for consumption*

Parameter	Concentration
Total Phosphorus	2 mg/l P (for 10.000 –100.000 Population equivalent) 1mg/l P (for > 100.000 PE)
Total Nitrogen	15 mg/l N (for 10.000 –100.000 PE) 10mg/l N (> 100.000 PE)
BOD <sub>5</sub>	25 mg/l O <sub>2</sub>
COD	125 mg/l O <sub>2</sub>
Total suspended solids	35 mg/l

*Table 3.5. Summary of directives for effluents parameters accepted in urban areas*

## Roadmap

Figure 3.8 shows the prioritisation of analytical targets for the water control in the beverage sector. Systems based on sensors already exist, and the interest for novel sensors and microsystems is to improve performances, reduce dimensions for more portable systems, and reduce costs, together with the integration of as much functionalities as possible in the same system.

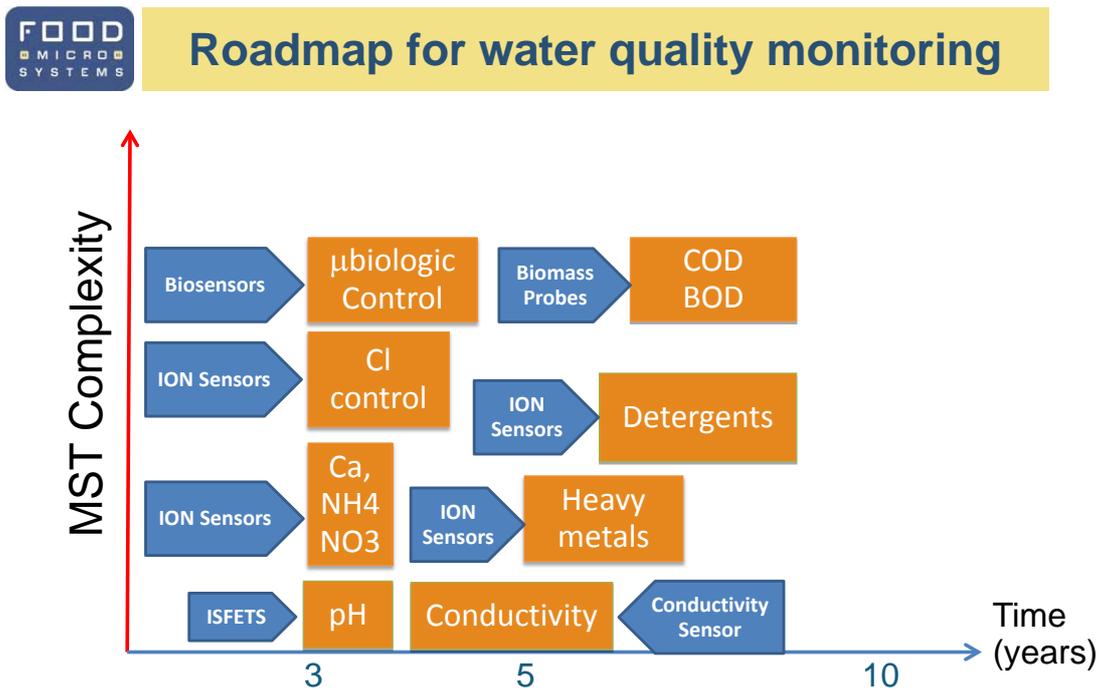


Figure 3.8: Roadmap of main parameters for monitoring water quality

## 4. Synthesis: Roadmap for the MST of interest for the beverage sector

In section 3, a set of wine and beer attributes and parameter has been identified for the key scenarios and critical points of their process. During the discussions carried out between winemakers and brewers and microsystems and instrumentation developers, the main parameters have been identified and the elaborators have shown an interest in new analysis systems. Such interest comes from different visions:

- the need of replacing current existing systems that are laboratory based, consume too much reagents or are not fast enough
- the wish for developing new systems that can help on carrying out new types of analysis that today are not possible.
- the wish for concentrating multiple analyses into a single system that can be portable autonomous fast and cost effective.

Together with MST researchers and developers, the capabilities of MST for addressing such challenges have been discussed, and an attempt to define the kind of systems that could solve each specific sensing requirement has been carried out. Solutions identified are of different complexity depending on the current degree of development. In some cases, micromachined sensors are available for other types of applications and can be tuned to the beverage sector requirements, giving a potential result t short term.

In other cases, the MST solutions require for a deep improvement of the performances of their components or building blocks or they require a big effort at the system integration level.

Finally, there are some other applications that will require complete new ideas, sensing materials and devices, and cannot be expected to be available at short or medium term. After this analysis it may occur that the microsystems are not the best solution, because of the impossibility to reach the specifications required for the application in the next years. However, even in this case, the exercise carried out can be of interest for driving more long term research of MST experts.

Globally looking at all types of MST devices identified in the different roadmaps of Section 3, we observe that a devices and systems can be classified in six main groups:

- gas and aroma sensors: volatiles, gases ...
- chemical sensors: pH, redox, ions, metals...
- bio-sensing systems: inmunosensors, genosensors, DNA...

- physical sensors: temperature, pressure, ultrasounds, turbidity, colour...
- RFID's, and other traditional electronics circuits.
- Artificial vision systems: CMOS cameras + image processing...

The last three groups are devices or systems that have already reached the level of research and development to be an industrial or quasi-industrial product, which means that they may be the basis of new short term implementations. Physical sensors, RFIDs and CMOS vision sensors are systems developed for other types of applications that are or can be very also very useful for the beverage industry.

This is not yet the case for the first three groups of sensors and systems. Despite the good research carried out in the last decade on chemical and biochemical sensors, in many cases they are not yet in the position of being fully reliable marketable products. In the case that they have achieved such level of development, they can be also improved and made more specific to the beverage sector requirements. These are the types of devices that will be studied in Deliverable 4.5 in order to identify what new materials, and technologies are necessary for developing real solutions for the beverage sector.

Table 4.1 presents the main targets that can be addressed with the different families of sensing devices and systems, and is a summary of the roadmap results shown in section 3 for the beverage sector.

It is observed that gas and aroma sensing systems may be of capital importance in the fast screening of raw materials and on the quality and authenticity of the final product and in the logistics sector.

On the other hand chemical and electrochemical devices are good candidates for on-line monitoring of the fermentation in the tank, of the maturation in barrel, of quality control in bottle and also for logistics, safety issues and water control monitoring via the detection of heavy metals and other important ions.

Another set of devices of potential interest are based on the bio-recognition of compounds in wine and beer: i.e. for safety, for water quality assurance but also during fermentation and ageing monitoring. In many cases, bio sensing methods despite they may show lack of high stability and short life time, may be used in the fermentation and ageing process because they show potentialities of developing new tests assays with fast response and small reagents consumption.

In Deliverable D4.5 a more detailed analysis of the technological roadmaps associated to the main families of devices identified here, will be presented. This will be the summary of what it is necessary from the sensors and microsystems point of view for the coming years for the food sector, according to the analysis carried out in the different application driven roadmaps.

Family of sensors	Main beverage targets that can be addressed with them
<b>Gas and Aroma Sensing systems (including e-noses)</b>	<ul style="list-style-type: none"> <li>• Aromatic Richness of grapes, barrels...)</li> <li>• TCA in wine and in cork</li> <li>• Phenolic Maturity of grapes</li> <li>• Authentication and Identification of wine and beer</li> <li>• Gases and vapours in the head space of tanks barrels and bottles</li> <li>• Multisensors in intelligent labels and RFIDs for logistics</li> </ul>
<b>Chemical sensing systems (including e-tongues)</b>	<ul style="list-style-type: none"> <li>• Aromatic Richness of grapes</li> <li>• Phenolic maturity of grapes</li> <li>• Authentication and Identification of wine and beers</li> <li>• Acidity of wine and beer at different stages</li> <li>• Polyphenol contents</li> <li>• Dissolved gas contents</li> <li>• Pesticides in end product for safety</li> <li>• pH, Conductivity, Redox potential</li> <li>• Detergents, Ions and Heavy metals detection in water.</li> <li>• Humidity in logistics</li> </ul>
<b>Bio-sensing systems</b>	<ul style="list-style-type: none"> <li>• Microorganisms detection in raw materials</li> <li>• Sugar contents at different stages of wine production</li> <li>• Anisoles and Phenols in barrels</li> <li>• Gluconic Acid in grapes and/or most</li> <li>• Malic and Lactic acids in tank during fermentation</li> <li>• Alterating microorganisms, amines, ochratoxines, allergens, brettanomyces, etc... at different stages of the production</li> <li>• Microbiologic control and COD of waste waters...</li> </ul>
<b>Other devices and systems: Physical parameters, Vision systems, TAGS</b>	<ul style="list-style-type: none"> <li>• Colour of grapes, and of wine and beer at different stages</li> <li>• Pressure, Turbidity, Temperature and Density during process and end product</li> <li>• Glass and barrel defects</li> <li>• Yeast viability during fermentation</li> <li>• RFIDs with sensors and intelligent labels for logistics</li> <li>• Particles in bottle for safety assurance</li> </ul>

*Table 4.1. Summary of attributes and parameters in the beverage sector that can be addressed by each family of MST devices of interest.*

## 5 Conclusions

European wine and beer producers are facing today challenging new issues due to the evolution of the consumption, changes in terms of the organization of the production structures, and increasing environmental concerns. The modern beverage industry needs tools for process control and quality assessment in order to better manage the main process steps, like the fermentation process. It is known that careful control of the fermentation process (considered one of the most important steps of the production process) can improve wine and beer quality and help producers to develop a product that satisfies evolving consumer tastes. However, as an example, despite being the cornerstone of the production process, fermentation is still controlled off-line today. Optimisation of fermentation monitoring and of the full process is mandatory and will add real value to European wine and beer producers by raising the quality standards and consistency of their products.

Sensors and microsystems may provide excellent solutions to complement laboratory analysis, by creating faster and portable systems for on-line and at-line use that will help on the full production process control of beverages. This better control may produce at the same time better products and at lower production costs.

Wine and beer are capital products for the European beverage sector because of their socioeconomic impact. To meet current challenges, the producers need new tools, devices, methods or services that can provide relevant information to orient their technical decisions, overcoming the costly and long-time current laboratory analysis. If analyses are made more efficient, wine and beer making management will be improved in the whole. New technical solutions based on MST leading to in-line/on-line monitoring of the quality attributes and safety indicators of beverages during production, going beyond simple measurements in the cellar of complex measurements in the lab, are of the utmost interest.

Developing new devices and smart systems for prompt monitoring following different (alternative/complementary) strategies such as miniaturised multisensing platform based on gas microsensors, gas and liquid micro-chromatography, and mid-infrared micro-spectrometry should be of interest for both the Food/Beverage and the MST sectors. Miniaturised systems based on MST should be flexibly adapted to different producers' needs and processing typologies in terms of cost and performance. Fast response, automatisisation and simplicity of use are other targets for microsystems solutions.

As reliable microsystems based devices and systems are not massively yet available for the beverage sector, an exercise of roadmapping the most promising solutions has been done within the FoodMicroSystems Project. Wine has been taken as a good

case study that has been extended to the beer sector when relevant. From the discussions of what should be measured, where, when and how, a complete set of product attributes, process parameters and potential sensors and systems for their control have been identified. They can be classified in some families of components and for the most promising a technological roadmap will follow. In Del 4.5.

The roadmap focus is set on on-site (in-line/on-line) process control. Depending on the technological suitability in-line/on-line will be prioritized, although even in the most challenging cases intermediate results may be valorised as affordable miniaturized bench-top automated analysers that will add an at-line additional control layer, close to the process, which is nowadays lacking in many small and medium production sites. These systems should be useful for not only the main process parameter (fermentation) but for the whole production chain.

To the opinion of experts that have participated in the roadmapping activity for the beverage products, the mid-to-long term MST impact on marketable products that Public Authorities expect from the R&D results is more prone to be reached in food/beverage process control (extended to the environment preservation) applications than in other Micro Nano Bio Systems (MNBS) fields. Here follows why:

- It is a tighter control in the early food and beverage production stages what minimizes economic losses, and it is an improved process control what brings deeper process knowledge and consequently a better product consistency and quality. Thus, whatever investment the producers may do to that respect will pay back sooner, easing the path towards implementation of any such approaches.
- Food and beverage safety, and health applications as well, are 'regulated' territories with mandatory sets of reference methods. New technologies are difficult to introduce in those scenarios. microsystems based safety systems may offer distinctive advantages, but if reference methods cannot be skipped in the end, this additional safety layer will be of dubious interest from a cost-benefit perspective, especially for food/beverage producers. Producers also have difficulties to convey positively these efforts to the consumer perception (they can be even counterproductive); consumers do not want 'safer' products, but 'safe' products: a safer version of a product may bring the idea to the consumer that the previous one was not safe enough.
- Quality control is less constrained by those issues. End product quality control is important and cost-wise from a company brand perspective. MST based systems may help allowing fast quality determination and product differentiation in terms of quality when possible. However, end product quality control will at some point mean discarding product and a negative economic impact. It makes sense to move quality control to the earlier stages of production, but this fact inherently points to the need of a better process control and new sensing tools.

- Microsystems may provide beverage producers with key information at critical check points in their process, supplementing their knowledge and experience, helping them to make the right decisions in pursuit of a product of quality. The higher degree of automation and higher level of information made available by those systems used in the main processing steps will contribute to a production of a more consistent quality homogeneity and optimised time to market.

In conclusion, the impact of microsystems will depend on their cost, savings on analytical reagents, response time and simplicity of use but especially on the value that beverage producers concede to the fact of having prompt information about their elaboration, helping them to take informed decisions, intervene and make corrective actions.

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## Annex: workshops organised for preparing the roadmap

The Roadmap development started with a series of workshops and bilateral meetings with representatives from various wine and beer elaborators, which gave as a result important information from the basic needs of monitoring the production process. These meetings were followed with two more specific to develop the roadmap. In those meetings not only the needs of the sector but also the potential for microsystems technologies, and their future acceptance were analysed. A first roadmap workshop was held in Vilafranca del Penedès, Spain and a second workshop was held in Paris, France.

In these specific face to face meetings different kinds of participants attended: sparkling and still winemakers, representatives of instrumentation manufacturers, companies that produce machinery for the beverage industry, and also active members of R & D groups (including researchers working on sensors and microsystems and also on oenology). Below, the agendas of these events hold in Spain and France are presented as well as the participants list of each meeting. The results of these meetings are shown in the various graphs in Chapter 3 of this report.

### **Meeting in Vilafranca del Penedes, March 11, 2013 (Organised by CNM-CSIC)**

The Penedès region, 40 Km approximately from the city of Barcelona is the most important in Spain for the production of Cava and it is the seat of important winemakers, along with the Rioja and Ribera del Duero.

The meeting went on to discuss in more detail the parameters of interest in the various stages of production of wines and champagnes and what type of sensors and miniaturized electronic systems were already being used and what others could be useful in the future if they improve the performances of the current systems or they are new systems for unresolved problems.

The steps to get to the necessary information were:

- to identify the need for the measurement of quality parameters or indicators needed at each stage of the process.
- to prioritize the sensors and systems that may be useful for monitoring these indicators, according to the needs of the wine producers and cellars not yet covered with commercial systems.
- To define the complexity of the solutions based on sensors and microsystems for the point of view of the technological complexity and availability. As a function of the two parameters (sector priorities and complexity of the solution), the feasibility of finding solutions to the problems is show graphically in two dimensional tables.

This methodology using Powerpoint is graphically presented in figure A1, and gave as a result, the diagrams already presented in Chapter 3.

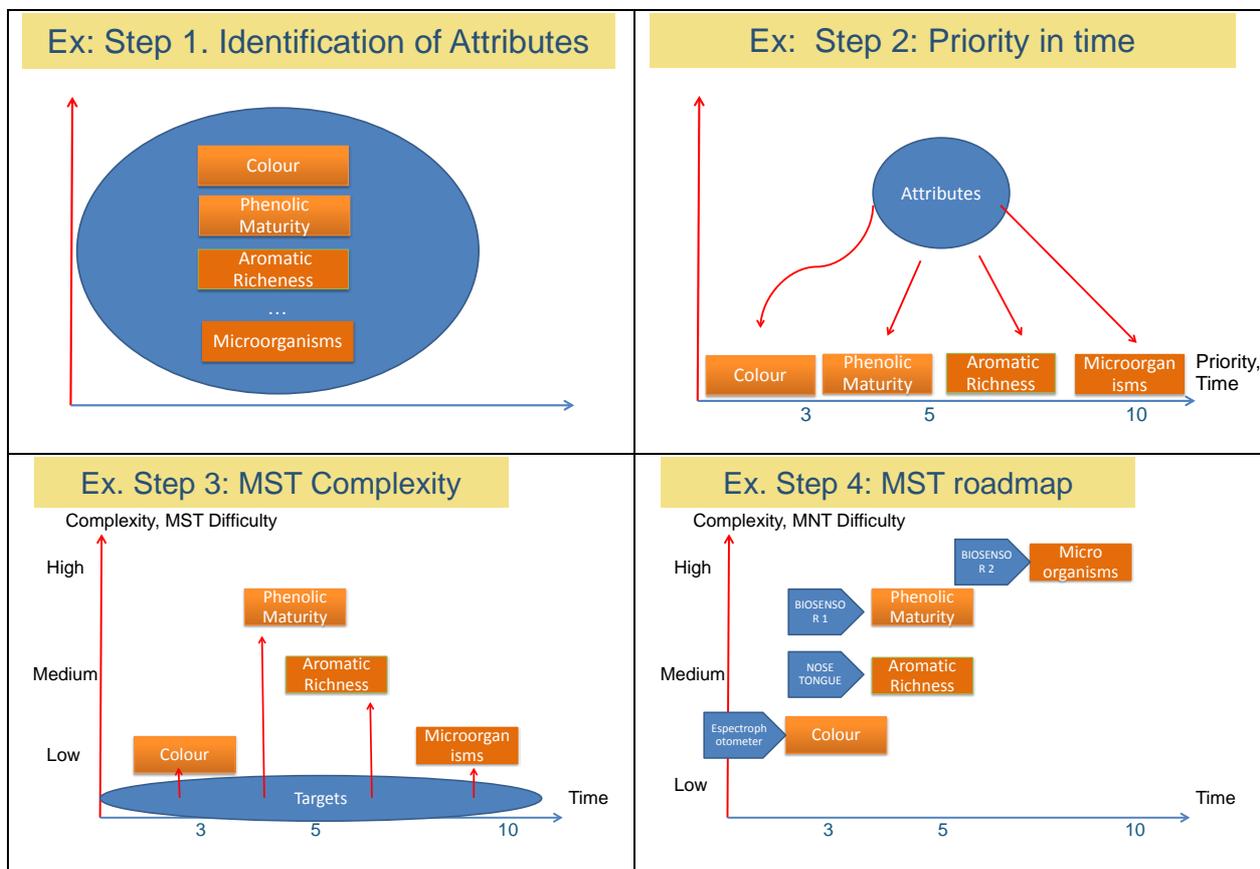


Figure A1 Methodology in four steps used during the discussions of the first workshop roadmap

Given the interest of all the representatives of the companies and research centres, participants preferred to participate in all discussions sequentially, so that parallel discussions were held with few actors were avoided.

Agenda:

**INTERNATIONAL EXPERT WORKSHOP  
MICRO-TECHNOLOGIES  
FOR THE WINE INDUSTRY**

How can Micro technologies and Smart Sensors help the wine sector to improve quality and safety, and reduce processing costs.

March 11<sup>th</sup>, 2013  
at INCAVI in Vilafranca del Penedès [Barcelona], SPAIN  
[www.foodmicrosystems.eu](http://www.foodmicrosystems.eu) / The FoodMicroSystems project is supported by the European Union (FP7/2007-2013) under grant agreement n° 287434.

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**Microsystems for the Food Industry, trends and applications.**

FoodMicroSystems is a project supported by the EU that started in September 2011. Its objective is to initiate the implementation of microsystems and smart miniaturized systems in the food sector by improving cooperation between suppliers and developers/users of microsystems for food/beverage quality and safety. In the food industry, microtechnologies can be used to micro-fabricate sensors and diagnostic systems. These technologies will significantly contribute to the economic benefit of European food industry by providing small and cost-efficient sensors and electronic systems with the added value of fast response for in-situ, on-line, or network solutions. In this way, it will contribute to an improved process control leading to overall food quality and, while reducing production costs, and strengthening consumer confidence.

**Expert Workshop "Micro technologies for the wine industry"**

The workshop is a follow-up of a previous event [Subirats, Barcelona, Spain, June 12<sup>th</sup> 2012] in which opportunities & challenges and barriers & drivers for the use of Micro-technologies for the wine industry were discussed. This workshop will bring together key players from the wine industry, equipment providers and specialists from the microsystems community with the objective of defining a roadmap for these applications.

As a coordination mechanism for the future, the roadmap is ensuring that demands of the wine sector are met by technological developments and will help future innovations in the sector.

If you are representing a company of the wine sector, by participating in the workshop you can direct the roadmap to goals that are of interest to your company and you will be able to initiate a collaboration and partnerships that can really benefit your companies.

If you are a researcher you will be able to show the maturity degree of your current developments and steer your future research in alignment with users' demands. In addition your inputs to the roadmap will be part of the conclusions sent to the European Commission for setting-up new R&D actions in the field of MNT for food in the Horizon-2020 Framework Programme [2014-2020].

**PROGRAMM**

**09:30**  
Registration

**10:00**  
Welcome and Tour de Table

**10:15**  
Objectives of the Meeting Defining R&D priorities on Sensors and Microsystems for the Wine Sector

**10:30-11:00**  
Overview of the FoodMicroSystems Project

**11:00-11:45**  
**TREND 1**  
Raw material reception  
Fast monitoring

**11:45-12:00**  
Coffee Break

**12:00-12:45**  
**TREND 2**  
Smart tanks (fermentation, yeast starters, storage)

**12:45-13:30**  
**TREND 3**  
Smart barrels (fermentation, maturation)

**13:30-14:30**  
Lunch Break and Networking

**14:30-15:15**  
**TREND 4**  
Smart bottles (fermentation, maturation, bottling, logistics)

**15:15-16:00**  
**TREND 5**  
Other on-line measurements (cleaning efficiency, organoleptic identification)

**16:00**  
Summary and Conclusions

**16:30**  
End of Workshop

**REGISTRATION**  
Participation for the workshop is free of charge, however, space is limited. Please register through our website [www.foodmicrosystems.eu](http://www.foodmicrosystems.eu)

List of attendees:

#	Name	Surname	Institution
<b>Winemakers</b>			
1	Xavier	Victoria	INNOVI
2	Vicenç	Segales	Codorniu
3	Laura	Tragant Capaz	Codorniu
4	Esmeralda	Payan Aguilera	Torres
5	David	Piqué Rosich	Gramona
6	Pilar	Urpí Bonell	Freixenet
7	Josep	Bujan Fernández	Freixenet
8	Toni	Cantos i Llopart	Juvé i Camps
9	Maria	Galup Torné	Covides
10	Simó	Serra	Castell de Perelada
<b>Instrumentation manufacturers</b>			
11	Antonio	Miró Vicente	BioSystems
12	Sònia	Viladevall Bou	BioSystems
13	Javier	Bengoechea	Biolan
14	Gustavo	Ghezzi	Ekinsa
<b>R&amp;D Oenology</b>			
15	Christophe	Cotillon	ACTIA
16	Santiago	Mínguez Sanz	INCAVI

17	Anna	Puig Pujol	INCAVI
18	Fina	Capdevila Mestres	INCAVI
19	Alejandro	Barranco	AZTI-Tecnalia
20	Sergi	Ferrer	Enolab-UV
21	Carmen	Berbegal de Gracia	Enolab-UV
<b>R&amp;D Microsystems</b>			
22	José Pedro	Santos Blanco	IFA
23	Santi	Marco Colás	IBEC
24	Mauricio	Moreno	UB
25	Eduard	Llobet Valero	URV
26	Cecilia	Jimenez	IMB-CNM
27	Manuel	Gutiérrez	IMB-CNM
28	Carles	Cané	IMB-CNM
29	Luis	Fonseca	IMB-CNM
30	Marc	Salleras	IMB-CNM
31	Manel	del Valle	UAB
32	Xavi	Cetó Alsedà	UAB
33	Jordi	Salazar Soler	UPC
34	Juan Antonio	Chávez-Domínguez	UPC
35	Kepa	Mayora Oria	IK4-Ikerlan
36	Deitze	Otaduy del Paso	Tekniker
37	José Antonio	de Saja	UVA
38	Luca	Francioso	CNR-IMM
<b>Other</b>			
39	Yvonne	Colomer	Triptolemos
40	Cristina	Peña	ACC1Ó

### **Meeting in Paris, April 2013 (Organised by Actia)**

At this second meeting in France, participants were mostly from the area of sensors and process-machinery producers than elaborators. In this event, thanks to the use of post-it labels of different colours a new iteration on the results of the Vilafranca workshop was done for each of the critical points, their attributes and the MST solutions of wine and beer elaboration. In figure A2 the photographs of various panels that focused the discussion of the participants are presented.



Figure A2: examples of panels used during the discussions of the Workshop of Paris

The meeting in Paris was also conducted following sequential sessions for facilitating the participation of all attendees in all discussions, so that the meeting was very fruitful too.

The brochure and list of attendees of the second workshop are shown below.

