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Description of the quality control and research laboratory of Viresol Ltd. Visonta, Hungary

In one of the most modern wheat processing plants in Europe, the high-quality production technology and the high level of quality required by customers when selling finished products require a high-quality testing laboratory background. Under this the mentioned requirements, the Quality Control and Research Laboratory was established and became operational in January 2019. The laboratory carries out quality control of raw materials, intermediate products and finished products related to production processes on a daily basis. The laboratory operates according to a continuous work schedule. The staff of the laboratory consists secondary, higher and scientific degrees and specialized qualifications employees. The laboratory analytical results are recorded in an SAP corporate governance system that ensures complete traceability throughout the production process.

One of the most important tasks of the laboratory is the batch testing of incoming raw materials, which is performed in a separate unit, the sampling laboratory. The sampling laboratory carries out a batch test of the incoming wheat raw material before intake, which quantifies mycotoxins (aflatoxin, ochratoxin-A, deoxynivalenol, zearalenone, T2/HT2), physical characteristics (hectolitre weight, miscibility, foreign materials, insect damaged seeds, broken seeds, chemical content) and basic ingredient parameters (moisture content, crude protein content, wet gluten content). Mycotoxins are quantified using the Neogen Reveal Q + max KIT system according to GIFSA/AOAC method. The physical tests are performed according to the methods of the MSZ 6383: 2017 standard, while the determination of the chemical properties is performed with a Perten IM 9500 device.

The central laboratory has been set up in accordance with the environmental requirements of ISO/IEC 17025: 2017. The laboratory did not yet have HAA (Hungarian Accreditation Authority) accreditation status during the time of writing of these paper, however the testing methods and procedures are based on international and domestic standards. The laboratory, built with excellent instrumentation and state-of-the-art materials, has been designed to perform the tasks expected in the future. With the completion of construction and construction work, a modern laboratory to meet the challenges of the 21st century began operations. The floor of the laboratory and the surface of the workbenches are made of acid and alkali resistant materials. Three chemical hoods have been set up for hazardous work processes, and there are also individual point air extractors in the laboratory. A chemical storage room were developed with individual air extraction and a separate poison depot store for the chemicals used in the laboratory. Used chemicals are temporarily stored in a hazardous waste storage room. For general analytical tests, analytical and tare balances, laboratory centrifuges, pH measuring instruments, conductometers, automatic pipettes, burettes, heating plates with magnetic stirrer, humidity measuring instruments with halogen-infrared heater and general glassware are available.

After receiving the bulk samples, the laboratory samples are prepared in a separate room. In most cases, preparation involves homogenization and grinding operations to achieve the appropriate particle size. The desired particle size of the samples is set with a Retsch ZM 200 laboratory grinder. A self-contained heating oven (Magmatherm MT1200) and a VWR Ventiline 180 prime airdrying oven for dry matter determination are available. In the sample preparation room, the particle size distribution of intermediate and finished products is determined on sieve systems for dry (Retsch AS 200) and wet (CISA BA200N) samples. The Merck-Millipore Rios DI-3-UV and Synergy UV water purification systems for the production of high-purity deionized water for laboratory tests are located in the sample preparation room also.

After sample preparation, the main nutritional parameters of the laboratory samples are also investigated in a separate room. Determination of protein content A Velp DLK20 automatic disintegration block supplemented with a unit for the absorption and neutralization of the generated acid vapors is used. The determination of nitrogen and protein content after digestion is performed by a Velp UDK 169 automatic distillation unit, which has an automatic sample exchange unit, which reduces the human resource requirement for the tests. The determination of the crude fat content of the samples is performed with a Velp SER 158, which is equipped with a solvent recovery unit, significantly reducing the amount of organic solvents used. To determine the crude fiber content, a Velp FIWE-6 apparatus is available for which a Velp Coex-6 type “cold extractor” was purchased for the preliminary, rapid degreasing of test samples with a higher fat content. The Faithful DZ1BCII vacuum oven in the laboratory allows fast and careful drying.

Much of the “classical” wet chemistry and instrumental testing takes place in the general analytical laboratory.

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The raw crop, intermediate products and finished products are tested according to a specific test procedure. Perten devices, which are also accepted in international practice, are used for wheat and flour tests (***Figure 1.***). Glutomatic are used to determine FN1000, gluten content, gluten index and water-binding capacity, while Chopin SDmatic devices are used to determine damaged starch content. Titrimetric tests are performed with Hach potentiometric titrators (Titralab1000) and a Karl Fischer volumetric titrator (KF1000). The sugar and starch contents of the test samples are determined with Anton-Paar instruments (Abbemat 300 refractometer, MCP5100 polarimeter), and the density and alcohol content of alcoholic samples are measured with an Anton-Paar DMA 4500 M instrument (MKEH-M1000765). The mycotoxin content of the finished products (aflatoxin, aflatoxin B1, ochratoxin-A, deoxynivalenol, zearalenone, T2/HT2) is determined using the Neogen Veratox ELISA kit.

A separate room has been set up in the laboratory for the chromatographic equipment in accordance with the requirements of the accreditation standard. Liquid chromatographic instruments have RI and UV/VIS detectors, gas chromatographs have FIDs. Fermentation and starch hydrolysis processes are monitored using Shimadzu liquid chromatography (HPLC) instruments. Shimadzu gas chromatographs are also used to examine the contaminants of intermediate and finished alcohol products and to qualitatively and quantitatively determine the denaturants added during the denaturation of alcohol (***Figure 2.***).

Atomic and molecular spectroscopic studies are performed in a spectroscopic laboratory located in a separate room too. A Shimadzu UV-1280 spectrophotometer is used for molecular spectroscopy. Atomic spectroscopic studies determine the content of macro-, microelements and heavy metals.

The analytical procedures are performed with an Agilent 4210 MP-AES atomic emission device (***Figure 3.***). The detection limit achieved with the device varies between 0.1-15 μg/L depending on the behaviour of the elements to be determined. Operation of the instrument does not require instrument gases used in atomic spectroscopy. The atoms of the sample are excited in microwave-generated nitrogen plasma. For plasma the required high-purity nitrogen is provided by a nitrogen generator device combined with a Mistral EVOlution compressor. The instrument gets nitrogen from the ambient air as plasma gas, reducing the cost of multi-element analysis and additionally eliminates the use of flammable acetylene and other expensive analytical gases. For precise and reproducible sample preparation for atomic spectroscopic studies, a Milestone Ethos Easy closed system microwave digester is used. In the device the digestion of samples takes place under pressure and temperature control. Using the equipment, the digestion time is more less, than the sample preparation process performed by the atmospheric process.

The microbiological unit of the laboratory consists of a general microbiological and sterile zone. The microbiological unit is ventilated through a HEPA filter system that provides sterile air. In the general microbiology department, sterilization processes (Raypa AES10 autoclave), preparation of test samples and determination of colony number are performed. In the sterile zone, samples are diluted, filtered, and inoculated in a Faster Safefast Classic 209 laminar flow cabinet. Rapid test procedures (3M Petrifilm) are generally used to determine microbiological contamination. To confirm the results obtained on Petrifilm, "classical" plate casting tests are performed in the laboratory. Memmert IN110 thermostat cabinets are used for battery number determinations.

The laboratory also has significant research and development activities. Within this framework, one of the most important programs is the optimization of maltodextrin production technology and the production of a product that meets the special needs of customers, which will be implemented within the framework of the GINOP-2.2.1-15-2017-00048 project. The use of maltodextrin in the food industry is on the rise in the European market. This product is mainly used as a stock enhancer in puddings, yoghurts, baby foods, but is also required by the brewing industry and is also used as an ingredient in muscle strengthening products. The uses of modified starch sold in the form of a moderately sweet powder are determined by the dextrose equivalent (DE) value, which characterizes the degree of degradation of the starch and can be adjusted by means of starch-degrading enzymes. The versatile use requires a flexible technological process in order to ensure the main property of the final product, the right sweetness and good solubility. To this end, the enzyme formulations with which maltodextrin production can be adapted to specific customer needs should be determined under laboratory conditions.

Another major experimental direction is the study of the retrogradation properties of starch. We are working on the development of a technology that can significantly reduce the tendency of post-crystallization (retrogradation) of edible wheat starch without the addition of other additives. Today, starch is used in many areas in the food industry, but due to the significant tendency to retrograde, the quality of the product containing starch is deteriorating. A good example of this is the waterlogging which appears on the surface of puddings. Currently, this tendency is most often suppressed by the use of additives that interact with the starch and suppress retrogradation processes during storage of the product. Sugars, polysaccharides such as gum-arabic are most commonly used for this purpose. These additives aren't harmful, of course, but they are not necessarily beneficial to humans. Hence it would be extremely important if the tendency of starch to retrograde could be reduced without additives and chemical modification. Several methods are known in the professional literature to delay retrogradation, but these are not yet used on a large scale. Our current development is aimed at the large-scale introduction of one such method, namely the mechanical treatment of starch. With the help of this innovative technology, the shelf life and quality of products that are sensitive to retrogradation can be greatly improved.

In addition to the mentioned above, research and development work includes improving the baking properties of the vital gluten product and – in the case of the native starch product – satisfies the individual customer. It requires enzymatic and viscometric application technology experiments.