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INPACTUS PROJECT: THE PATH TOWARDS A FOREST-BASED BIOECONOMY

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SUMMARY

The inpactus project, promoted by The Navigator Company, RAIZ, the University of Coimbra and the University of Aveiro, in close collaboration with other institutions, represented the largest investment in Portugal in R&D in forest-based bioeconomy. The project was run between 2018 and 2022 and the activities were focused on new products, process improvement and new technologies, in the areas of pulp, environment, UWF and packaging paper, tissue and bioproducts based on eucalyptus. The project has generated 40 patent applications, about 150 publications (among all partners) and 4 new products. One of the activities with high impact for the The Navigator Company was the technical support to high-yield pulp production for a new line of packaging paper, gKRAFT, launched in the end of 2021. Additionally, research in Tissue paper resulted in three innovative tissue products: Amoos Naturally Soft™, Amoos Air Sense™ and Amoos Aquactive™. Many other well succeed examples of applied research and figures will be highlighted. Besides the scientific and technical achievements, the project led to important outcomes for the future of Forest-based Bioeconomy in Portugal, as the training of highly qualified human resources, new capabilities in forest-based bioproducts (pulp and paper included) and a long-term University-Industry partnership.

Keywords: inpactus, bioeconomy, bioproducts, forest, pulp, paper, tissue

INTRODUCTION

The inpactus project arose from the initiative of The Navigator Company, in partnership with Universities and Research and Technology Institutes, to develop a university-industry excellence platform of a multipolar nature, in innovative products and technology based on eucalyptus.

In 2018, FEDER funding was approved as part of the Competitiveness and Internationalization Operational Programme, in the Incentive System for Companies with a consortium composed by The Navigator Brands, The Navigator Pulp Aveiro, Forest and Paper Research Institute - RAIZ, the University of Coimbra and the University of Aveiro, in partnership with other renowned institutions – Figure 1-A.

The project, structured in 15 activities, was developed over 4 years and 9 months, with a global investment of 14.6 million euros and an eligible investment of 13.3 million euros. Its operation relied on a team of around 180 people, including 24 PhD students and 2 invited Chairs, Paper Science and Biorefinery.

The scope of the project was to develop new knowledge on forest-based bioproducts, improving both processes and products in the already consolidated business areas of The Navigator Company, always keeping environment and sustainability as a priority. Additionally, the project aimed to develop new bio-based products by applying or developing new technologies for the forest sector, based on eucalyptus, in the perspective of business diversification. In an increasingly competitive and demanding market for the sector, this was a take-off project to create new business opportunities and simultaneously has provided knowledge to promote better process and product performance, some of them have already been put into practice.

Figure 1 – inpactus partners: 3 Companies, 4 Research Institutes and 6 Universities

HIGHLIGHTS ON PULP PRODUCTS AND PROCESS

The growing concern with the supply of wood was the driving force behind one of the activities with the greatest impact on the sector. Different eucalyptus and non-eucalyptus species were evaluated individually and combined to obtain the best kraft pulping conditions to maximize pulp yield and performance through bleaching. Several strategies were evaluated, such as individual impregnation and optimization studies regarding temperature, time and alkaline charge. Another strategy was based on pulping additives, evaluating the dosage and the effect in eucalyptus mixtures pulping. For some pulping additives, applied under optimal conditions, the yields were improved 2.3% for the *Eucalyptus globulus/urograndis* mixture and 1.9% for the *Eucalyptus globulus/nitens* mixture, also observing a decrease on kappa number for some conditions.

Regarding bleaching response for the sequence $D_0EpD_1D_2$, *E. globulus* pulps with kappa number ~15, showed better bleachability than those produced with lower temperature and longer pulping time (constant H factor) while the *E. urograndis* pulps showed an opposite trend. For pulps with kappa number ~18, after the oxygen stage, the best bleaching response (sequence $(O/O)D_0EpD_1D_2$) was obtained for pulps resulting from higher temperature and shorter cooking time for both *E. globulus* and *E. urograndis*. Mixture pulps exhibited better bleachability at intermediate pulping conditions – Figure 2. Comparing the results of the bleachability with and without the oxygen stage, applied to pulps with kappa numer~18 and ~15, respectively, it can be inferred that the O/O stage lightened the differences between pulps produced at different pulping conditions, with the additional benefit of higher viscosity.

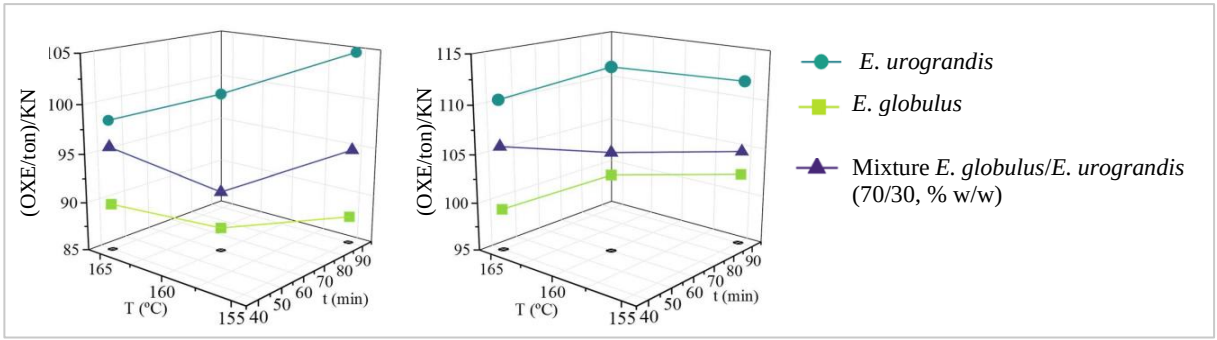


Figure 2 – Oxidation equivalents per ton of pulp, divided by the kappa number (OXE/ton)/KN for *E. urograndis*, *E. globulus* and mixture pulps produced at different kraft pulping conditions for a H-factor constant.

The process to produce High-Yield Eucalyptus Kraft Pulp (HYKEP) (kappa number 50-70) of *E. globulus* was developed. This innovative process makes it possible to reduce the specific consumption

of wood by more than 20%, compared to conventional pulping (to kappa number 14-16), and to produce packaging paper. This result was strategic for the Company, as it highlighted the excellent suitability of *Eucalyptus globulus* for packaging paper. A whole new family of packaging papers was thus created, the gKRAFT, launched in 2021. In a further stage of R&D related with the production of high-yield pulps, the results were described in a provisional patent application.

Still in the fiber line of the pulp mill, a modified bleaching sequence was developed by the introduction of an enzymatic stage, with the objective of decreasing bleaching costs. The optimised $OXD_0EPD_1D_2$ sequence reduce ClO_2 and NaOH requirements in the D_0 and Ep stages by 20 and 10 %, respectively, in comparison to the conventional bleaching sequence. The additional advantage was the decrease of AOX amount in the D_0 stage filtrate by up to 30 %. Furthermore, the brightness stability of bleached pulp was improved, and the papermaking properties were maintained. Although the process is not yet implemented, the results allowed to highlight new opportunities in the pulp mill by using enzymatic-aided beaching, keeping the awareness on efficient enzymatic commercial products.

In the scope of inpactus, the process verification and decision support tool "Navigator Process Check" developed provide support to process engineering for solving problems and optimizing pulp cooking, bleaching, washing, and screening processes. Models were developed based on data from the equilibrium of non-processual elements and integrated into process simulation tools, which are currently being used in the Company to support the optimization of industrial pulp production, including the use of water resources.

Other relevant outcomes resulting from R&D in PULP and PROCESS are briefly pointed out:

- Design and conception of a laboratorial equipment and procedures for producing air-laid sheets for the development of R&D on non-wovens based on eucalyptus fiber and bio-based polymers. This work aiming to open new perspetives for BEKP applications, resulted in two provisional patent applications.

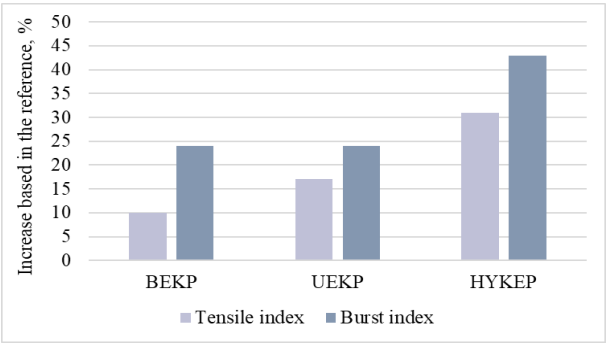
- The aplication of biomass fly ash, collected at biomass boiler in pull mill, as secondary raw material in a commercial screed mortar formulation, as filler or as a cement substitute was proved. To maximize the compatibility of the ash with these applications some pre-treatment processes, such as milling and sieving, were tested and applied. This work has demonstrated the potential, the limitations to be overcome and the solutions for the application of a (still considered) residue in the production of more sustainable screed mortars.

HIGHLIGHTS ON UWF PAPER AND PACKAGING

One of the areas of potential application of microfibrillated celluloses (MFCs) is the use in paper production. MFCs have been investigated due to their potential as strengthening agent in paper production. The main objective was to study the use of MFCs as an additive to increase its internal resistance, without impairing the drainage and finding a balance with the desired increase in filler content (retention).

MFCs were produce from different pulps including bleached eucalyptus kraft pulp (BEKP), unbleached eucalyptus kraft pulp (UEKP) and HYKEP, and their performance and the energy required for the production were evaluated. The incorporation of 5% of MFCs produced from BEKP, UEKP and HYKEP in the respective pulps, including retention agent, led to an increase in mechanical resistances, in particular burst and tensile indexes, reaching to an increase of over 40% for HYKEP – Figure 3.

The next steps are addressed in a new Project, From Fossil to Forest – Sustainable packaging and products to replace fossil plastic, supported by Recovery and Resilience Program, which will be running until 2025. In the scope of the new project, pilot and industrial trials will be performed to optimize both the production and the incorporation of CMFs in packaging paper to promote the mechanical strength, required to some applications.



Microfibrilated cellulose produced from HYKEP at inpactus project

Figure 3 – Burst index and tensile index, in increment relative to the reference (respective pulp with no CMF) for CMF produced from BEKP, UEKP and HYKEP, incorporated in respective pulps at 5% and evaluated in handsheets.

Other activity was related with cationization of starch, providing insights on the feasibility of on-site production. The optimization of the cationization process was performed on commercial native starch, reaching different degrees of substitution. The tests of incorporation in papermaking and evaluation of paper properties shown a similar performance to commercial starches. Also, in the topic of papermaking additives from renewable sources, a soluble CMF was produced by introducing cationic groups. The aim was to replace, at least partially, the cationic polyacrylamide (CPAM) used in papermaking. Based on the obtained results, filler retention and mechanical properties, as well as sheet formation, should be improved to reach the performance of the commercial CPAM. Despite this, the potential of cellulose derivatives as additives in papermaking has been demonstrated and future steps have been addressed for a new range of bioflocculants.

Many other results should be emphasized, due to their innovative character and importance for the paper and packaging sector, and some highlights are:

- Sensors in paper, with potential application in security papers, traceability, anti-counterfeiting, or other high-added value applications, were developed and applied.

- A methodology for characterizing the fiber orientation in the ZD direction of paper was developed along with a roadmap to diagnose the causes of curl-related problems.

- New formulation for paper surface treatment aiming to improve the printing quality.

- A Life-Cycle Assessment of UWF paper production for the Company mills was performed, identifying the impacts and the opportunities for improvement. The obtained carbon footprint was compared with other tools developed for the sector, as a validation process of the overall conclusions.

HIGHLIGHTS ON TISSUE PAPER

In the inpactus project, potential alternative source of cellulosic fibers for tissue production were tested. Tissue handsheets were produced by incorporating UEKP and unbleached pulp produced from eucalyptus bark, and the tissue properties were evaluated. The studies revealed that incorporation of bark pulp have improved strength properties such as tensile and tear, important for the performance of the end-product and for paper machine runnability. Additionally, higher bulk, tensile index and water absorption were achieved, with no significant variation in softness. The incorporation of UEKP had led also to encouraging results. Following the successful industrial trial, a new tissue product using unbleached eucalyptus kraft pulp was developed and launched, the Amoos NATURALLY SOFT™ – Figure 4-A. This work has resulted in two patents.

The functionalization of tissue paper by using sustainable additives was also in the scope of the inpactus project. It included active agents such as essential oils, fragrances and/or surfactants, in the form of emulsions and/or microcapsules. The formulation of these additives and the production of emulsions and microcapsules was investigated and optimized. The active agents were applied on paper tissue,

obtaining differentiating properties, such as controlled and prolonged release of aroma, and/or sanitizing action. The knowledge transferred to the Company led to the launch of new tissue products: Amoos AIRSENSE™ with prolonged aroma release, and Amoos AQUACTIVE™, the new tissue product for cleaning surfaces, with water activated foam – Figure 4 - B and C – leading to the expansion of The Navigator Company's product portfolio.

Figure 4 – New Tissue products launched by The Navigator Company resulting from inpactus R&D

Many other outcomes from R&D in Tissue in the scope of inpactus are worth mentioning:

- Evaluation of the effect of different enzymes as refining aids and the impact on tissue properties to support the Company in the selection of enzymatic products and in the adjustment of refining conditions.

- Development of a simulation tool, the *SimTissue*, for correlations between the tissue paper process inputs and the end-use paper properties. The tests were performed with case studies with industrial interest, using different fiber mixtures, fiber modification treatments, micro/nano fibrillated cellulose, and biopolymer formulations, to estimate tissue softness, strength, and absorption properties.

- Development of a laboratorial embossing system to simulate the operation with different patterns and revealing the optimal conditions of pressure and rubber hardness that give better tissue properties.

- Conception of a tool designed for load optimization for transporting hygienic products.

- Life-Cycle and Sustainability Analysis was carried out for tissue production according to various scenarios, providing data to support decisions and communication initiatives.

HIGHLIGHTS ON BIOPRODUCTS AND BIORREFINERY

To complete the scope of forest-based bioeconomy, the inpactus project also addressed processes, technologies, and new products based on eucalyptus residual biomass and on BEKP, taking advantage of the integration in pulp and paper mills. Two dozen topics were studied, in different knowledge areas, comprising bioactive compounds, biomass deconstruction technologies for sugar production, processes for ethanol and for bacterial cellulose, thermochemical processes (gasification and pyrolysis technology), separation technologies for lignin, new products from lignin and new high-added value products from BEKP. Figure 5 presents the overall perspective of biorefinery processes integrated in a pulp mill, and the materials and products that can be generated, regarded as new business opportunities for the forest-based sector.

Bioactive extracts were obtained from forest biomass through an integrated process, involving sequential extraction steps and reuse of the extraction medium. Bioactivity studies proved that the extracts have a neuro-protective action, opening the perspectives to prevent several pathological cellular processes and to improve cognitive function, as Alzheimer's disease retardant. Other important activities are antioxidant, anti-inflammatory and anti-aging effect on the skin. Taking advantage of this bioactivity, the extracts were incorporated as part of a formulation for a facial cream or embedded in a bacterial cellulose mask. The bacterial cellulose was produced from cellulosic sugars. The approach has resulted in the development of an entirely bio-based material with bioactive and adequate mechanical properties for potential anti-aging skin care applications. Resulting from this work, two provisional patent applications were filled. In parallel, a process based on supercritical extraction, to obtain extracts with high selectivity for some interesting compounds, was developed and optimized.

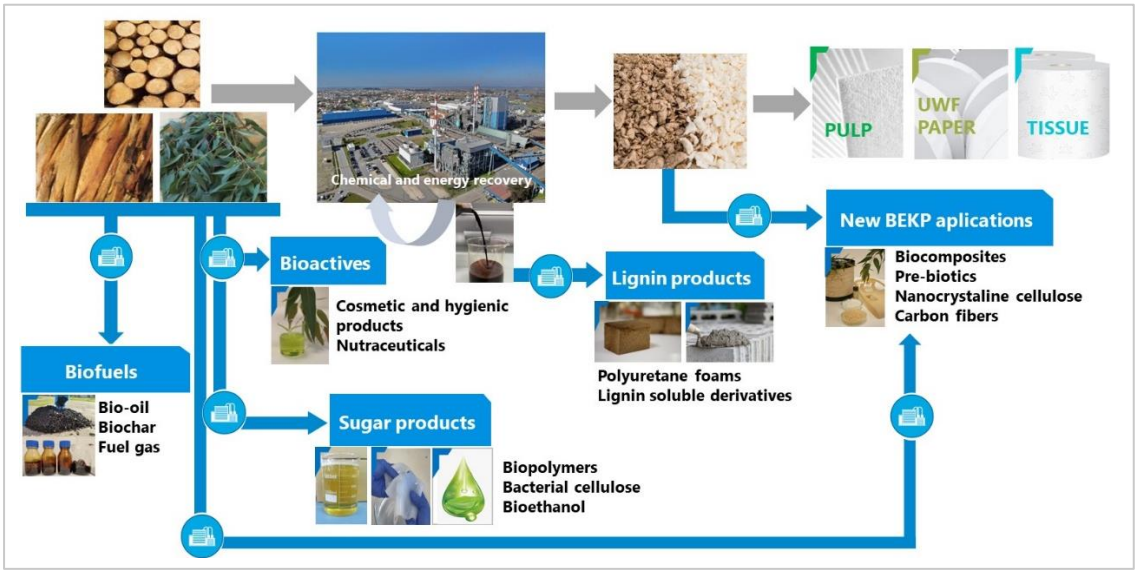


Figure 5 - New opportunities for the forest-sector: bioproducts generated from biorefinery processes integrated in the P&P industry addressed by inpactus project.

The next step is already ongoing with the operation of a pilot unit for extraction (essential oil, as well as water and ethanol soluble extracts) – Figure 6. The objective is to upscale the process and optimize conditions, the biomass storage time and processing, to consolidate the techno-economical assessment. After optimization, in the pilot trials, essential oil production has reached 19 L per ton of biomass (dried weight) and the triterpenic compounds extraction yield was about 14 kg per ton of biomass (dried weight). In both cases, the yields represented good perspectives for the business case.

Figure 6 – Preparation of biomass, pilot unit for steam distillation and essential oil produced

Many other relevant outcomes arose from the work developed in inpactus project, such as:

- The gasification of eucalyptus biomass was studied in a pilot unit (80 kWth), builded during the project, demonstrating flexibility to adjust the conditions according to the quality required for the gas. One of the conclusions was that the use of O_2 or steam improved the gas quality in terms of H_2 concentration, H_2:CO molar ratio, and lower heating value of the dry producer gas, showing the potential of this strategy to overcome some limitations of the conventional air gasification technology, generating relevant data to support the scale-up of the process.

- Demonstration that *E. globulus* bark and primary sludge are suitable raw materials to produce cellulosic sugars and bioethanol. The fed-batch strategy was identified as being the one that results in hydrolysates with higher sugar concentrations (136 g/L of glucose and 25 g/L of xylose), which can be converted to ethanol in a concentration 76 g/L, representing a yield of 84%.

- Development of bio-polyols with a content of lignin (isolated from black liquor by LignoBoost® process) greater than 20% and it use in the production of rigid polyurethane foam and addesives.

- Establishment of a methodology for the cationization of lignin and consequent LignoBoost® kraft lignin solubilization at concentrations up to about 500 g/L regardless of the pH. This lignin derivative was tested in several applications, such as flocculant and as cemmet additive.

- Application of BEKP in the process for nanocrystaline cellulose prodution. A new anisotropic phase was identified, as well as a new indirect method that shows the interactions between nanocrystaline cellulose and water, openning the possibility of innovative applications. In this scope, a low-cost anti-fog coating was developed and patented.

- Development of a process to produce xylooligosaccharides, a prebiotic, from BEKP. Prebiotic activity has been proven, showing activity similar to a commercial product. The reintroduction of the treated pulp into the mill was studied, concluding that the process did not change the characteristics of the pulp. Two patents as resulted from this activity.

- Demonstration of the high potential of composites based on renewable matrices (for example with polylactic acid), reinforced with micronized BEKP fibers up to 40% and using additives of renewable origin, for replacing fossil-based ones. The work has continued with the installation a pilot unit (pre-mixer and extruder unit) for scale-up. This topic is currently addressed in the Agenda From Fossil to Forest – Sustainable packaging and products to replace fossil plastic, supported by Recovery and Resilience Program.

CONCLUDING REMARKS AND FUTURE

For the topics with more relevant developments and with higher business potential, a technical-economic evaluation, modelling and simulation of processes, or scale-up has been carried out. The objective is to increase the TRL and to provide additional data for process flowsheets, allowing the mapping of costs and economic assessment. This work is currently ongoing.

For the topics closer to the actual core business, in particular the outcomes related with process, pulp production, packaging, and tissue paper, with no major requirements for adaptation of industrial assets, the outputs are being endogenized by the Company. Some of them have already resulted in new products for the market, such as gKRAFT and the three innovative tissue products. Other topics, such as packaging and biocomposites are now continuing their R&D and Industrialization path in the scope of the Agenda From Fossil to Forest – Sustainable packaging and products to replace fossil plastic, supported by Recovery and Resilience Program. Additionally, in 2023, the first National Co-creation Program in Forest-Based Circular and Digital Bioeconomy was launched, and the opportunity was taken to promote the collaboration between small companies, start-ups and inpactus work groups.

The project has generated so far 40 patents (granted or provisional), 4 new products and more than 70 prototypes and informatic tools, about 150 technical or scientific publications and more than 200 participations in conference. Besides the knowledge and the scientific advances, the project strengthened the future of Forest-based Bioeconomy in Portugal, by training of highly qualified human resources, 24 PhD students and 45 MSc, and supporting new capabilities in forest-based bioproducts (pulp and paper included). The inpactus project is an exemplary success case of University-Industry partnership.

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