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Smart tags v1

D3.5

DigInTraCE

A Digital value chain Integration Traceability framework for process industries for Circularity and low Emissions by waste reduction and use of secondary raw materials

Funded by the European Union

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Dissemination level	Public (PU)
Type of deliverable	R – Document, report
Work package	WP3 – Sensing, Sorting Technologies and Process
Deliverable number	D3.5 Smart Tags v1
Status - version, date	V1.0, 24/06/2024
Deliverable leader	VTT
Contractual date of delivery	30/06/2024
Keywords	smart tag, 2D bar code, sensor, indicator, humidity, temperature

Quality Control

	Name	Organisation	Date
Peer review 1	Francisco Javier Real Salas	CIRCE	17/06/2024
Peer review 2	Alexandros Chatzipavlidis	ICCS	20/06/2024

Version History

Version	Date	Author	Summary of changes
01	03/21/2024	Liisa Hakola	First draft of document structure
02	17/06/2024	Liisa Hakola, Elina Hakola, Kaisa-Leena Väisänen	Final document for internal review
03	24/06/2024	Liisa Hakola	Final deliverable with internal review comments addressed

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List of abbreviations and acronyms

Abbreviation	Meaning
2D	two-dimensional
1ME2PRO	1-methoxy-2-propanol
BCG	Brom Cresol Green
IoT	Internet of Things
NFC	Near Field Communication
PE	Poly Ethylene
PET	Poly Ethylene Terephthalate
PP	Poly Propylene
PVP	Poly Vinyl Pyrrolidone
QR	Quick Response
RFID	Radio Frequency IDentification
WVTR	Water Vapour Transition Rate

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Executive Summary

This deliverable is a report on smart tags development for two DigInTraCE demo cases, namely wood composites in Greek demo case and polyester textiles in Belgian demo cases. Firstly, this deliverable provides scientific and technical background on smart tags and their components: data carriers and technologies for condition monitoring. Besides, all the different alternatives showcased in the deliverable are compared.

In the experimental part the deliverable describes the design and development of the smart tags for the two demo cases involving humidity and temperature sensing smart tags, respectively. The materials and methods used for smart tags development are described followed by the results.

Finally, the next steps and recommendations for future development are outlined. It is concluded that the humidity and temperature sensors are ready for integration into the demo cases, and suggestions are given for fine-tuning the technologies. The next activities will be to investigate data integration and integrity, carry out stakeholder engagement, and develop a formaldehyde sensing smart tag for the Greek demo case.

1 Introduction

1.1 Project intro

The DigInTraCE project aims to create a transparent and interoperable Decentralized Traceability platform by employing innovative tracking, sensing, and sorting techniques. Emphasis is placed on dynamically updating Digital Product Passport (DPP) schemes to support certification and quality validation. Additionally, the project integrates AI-based decision-making mechanisms to optimize processes and lifecycles. DigInTraCE seeks to enhance the utilization of secondary raw materials through up-cycling, reuse, and upgrade technologies. The project also contributes to standardization efforts, ensuring open and easily accessible data. Exploring new business models, DigInTraCE aims to create economic opportunities, promote digital skills, and address regional social needs.

Driven by six objectives, DigInTraCE outlines its trajectory. The first objective (O.1) involves designing and implementing solutions to optimize the utilization of secondary raw materials and minimize waste within circular value chains. The second objective (O.2) focuses on developing and demonstrating innovative concepts for material tracing through a decentralized digital platform, facilitating the tracing and certification of secondary raw materials. The third objective (O.3) entails delivering cutting-edge real-time sensing and sorting mechanisms to enhance data exchange through a dynamic DPP. Additionally, the fourth objective (O.4) aims to improve accessibility to crucial material data by utilizing smart tags, smart contracts, open software, and immersive technologies. The fifth objective (O.5) centers on validating the efficacy of DigInTraCE technologies across four distinct value chains. Finally, the sixth objective (O.6) involves empowering local and regional entities by actively involving them in developing educational resources for workplaces and educational institutions. This collaborative effort fosters the adoption of DigInTraCE solutions within the broader community and facilitates knowledge transfer to maximize the project's impact.

1.2 Purpose of the deliverable

Deliverable *D3.5 Smart tags v1* is a report on activities carried out in *Task 3.3 Smart Tags for real time sensing (T3.3)* until M18. This deliverable exhibits Smart Tags use scenarios, presents tested real-time sensing and identification technologies and related results, presents approaches to support data integrity and integration, as well as an initial overview description of stakeholders' engagement strategies (in Conclusions, as next steps). This is the first version of the deliverable, which is focused on selected smart tags use scenarios to support demo case implementation (Greek and Belgian demo cases), and on experimental tests for the development of humidity and temperature sensing smart tags. Furthermore, the final version (at M36) will complement this deliverable including aspects for integration of data spaces with the smart tags, and report on the stakeholder engagement activities carried out.

1.3 Intended audience

The dissemination level of D3.5 is Public. Therefore, D3.5 on smart tags technologies aims for widespread dissemination and adoption across sectors, targeting diverse audiences, including academic and research community, industry and business sector, policy makers and regulatory bodies, broader society, etc. By engaging these groups, the deliverable aims to promote smart tags technologies, encourage sustainable practices, and foster innovation across sectors. The intended audience of the current document is mainly the DigInTraCE consortium, specifically the partners leading and participating in Greek and Belgian demo cases. Furthermore, partners developing digital product passport in *WP2* and *WP4* will find the report useful from data carrier and data spaces perspectives.

1.4 Structure of the deliverable and its relationship with other work packages/deliverables

In this deliverable, Section 2 describes the smart tag concept and relevant scientific and technical background. It also describes the smart tags concepts selected for the Greek and Belgian demo cases, and associated development goals. Sections 3 and 4 report the results for humidity and temperature indicator development, respectively. Section 5 summarizes the results, outlines the next steps for the completion of *T3.3*, and identifies recommendations for future development.

This deliverable is linked to requirements of Greek and Belgian demo cases that are being developed and integrated in *WP6 Piloting and Demonstration*. The smart tag concepts presented in this deliverable have been tailored to meet the data exchange and monitoring requirements from these demo cases. Furthermore, inclusion of smart tags as data carriers into digital product passports (DPPs) is being developed in *WP2 Holistic framework for digitalization of circular value chains* and *WP4 Digital tools and platform development*. Smart tags and standardized data spaces have been described briefly in *D2.4 Digital Product Passport Concept v1*. *D3.6 Smart tags final* will be a follow-up of this deliverable and will report activities carried out in *T3.3* during M19-M36 as highlighted in Section 5.

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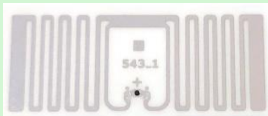
2 Background

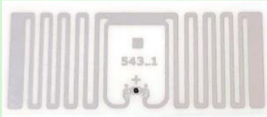
2.1 Smart tags

Smart Tags are visible or electronic markers with environmental sensing functions combined with software intelligence (machine vision, user information, location, etc.) (Gligoric et al, 2019) (Hakola et al, 2021). These features provide context-aware services to the end users and enable their connectivity to the Internet of Things (IoT). The smart tags consist of (1) a data carrier that provides item-level identification and access to data stored into databases in the cloud, and (2) a sensing solution that monitors selected environmental or product conditions during product lifecycle.

The most typical data carriers used in smart tags are 2D (two-dimensional) barcodes, RFID (Radio Frequency IDentification) tags and NFC (Near Field Communication) tags. The most common 2D barcodes include QR (Quick Response) codes and Data Matrix codes. They consist of black and white squares i.e. cells. A sophisticated error correction algorithm is included, which means that information is readable even if up to 30 % of the code is destroyed. 2D barcodes are readable with smartphones and several other camera-based technologies. RFID uses electromagnetic fields for identification and tracking. Passive RFID tags are powered by the radio energy transmitted by the reader. Active RFID tags have a battery to transmit its ID signal regularly. Battery-assisted RFID tags contain a battery but are activated only in the presence of a reader. RFID tags can be read with a dedicated RFID reader. NFC tag is a short-range, smartphone-readable version of RFID. The different data carrier technologies are compared in Table 2.1.1 (Tenhunen-Lunkka et al, 2022) (Hakola et al, 2023). For the DigInTraCE project, 2D bar codes have been selected due to their affordability and capability of visual monitoring.

Table 2.1.1 Comparison of data carrier technologies.

Technology	**2D barcodes**	**RFID** 	**NFC** 
Availability	Open and proprietary technologies	Commercial tags available for a variety of frequencies and reading distances	Commercial tags available for 13.56 MHz frequency
Price level	Low	High	Medium
Manufacturing	Printing	Electronics processing, printing	Electronics processing, printing
Reading	Visual reading, distance ca. 3-30 cm	Electromagnetic reading, reading distance up to several meters	Electromagnetic reading, distance 0.5-3.0 cm

Technology	2D barcodes	RFID	NFC
			
Physical size	Scalable, min. ca. 1 cm square (reading requirement)	Scalable, typically starting from 1 cm	Scalable, typically starting from 1 cm
Information capacity	Up to 7000 characters	Up to MBs	Up to MBs
Expected lifetime	Several years	A few years	A few years

Monitoring and sensing technologies enable access to dynamic information and its meta-information content due to the changing environmental conditions when reading the codes. The main technologies are functional inks, sensors, and visual indicators as described and compared in Table 2.1.2. Functional inks react with a reversible or irreversible visual colour change to changes in the surrounding conditions. Sensors are devices that detect and respond to some type of input from the physical environment, and the output is generally a signal that is converted to a human-readable display (Chansin, 2015). Indicators are sensors based on optical reading, such as colour change (Sipiläinen-Malm & Hurme, 2008) (Ghaani et al, 2016). In the simplest form, they are functional inks reacting to environmental conditions, but often require some activation step before usage.

Table 2.1.2 Comparison of sensing technologies.

Technology	Functional inks	Sensors	Indicators
Principle	Visual colour change, reversible or irreversible	Response with a signal, logging	Visual colour change, irreversible
Availability	Commercial inks	Commercial technologies, developmental technologies	Commercial technologies, developmental technologies
Parameters to monitor	Thermochromic, photochromic, fluorescent, phosphorescent, hydrochromic inks	Biosensors, capacitive sensors, piezoresistive sensors, piezoelectric sensors, photodetectors, temperature sensors, humidity sensors, gas sensors	Time temperature indicators, oxygen and integrity indicators, freshness indicators
Price level	Low	Medium to High	Low to Medium
Manufacturing	Printing	Electronics processing, printing	Printing, coating, activation
Compatible data carriers	2D barcodes	RFID, NFC: sensors integrated in chip or stand-alone sensors	2D barcodes, RFID, NFC

Functional inks and indicators were selected for DigInTraCE since those are compatible with 2D bar codes, and possess properties important to demo cases, such as affordability, visual reading, and availability of several different parameters to monitor. Visual monitoring technologies are available for monitoring the following parameters, either commercially or as developmental versions from VTT (marked with (VTT)):

- Temperature
- Exposure to light
- Time and temperature
- Humidity (VTT)
- Leakage (oxygen) (VTT)
- Volatile compounds: H2S, aldehydes, ketones, amines, ethanol, nitrogen (VTT)
- Ethylene (VTT)
- Authenticity

2.2 Smart tag concepts in DigInTraCE

Smart tag concepts were developed for two demo cases of the project:

1) Greek demo case: wood composites
2) Belgian demo case: polyester textiles

For the Greek demo case the development goals were to:

1) Formulate a humidity sensing smart tag compatible with plastic substrate. The threshold humidity of 5-70% was targeted at.
2) Attach the smart tag on wood surface in a way that allows monitoring the humidity of the wood – not the surrounding atmosphere.

To achieve (1) a humidity irreversible indicator concept reported by Hakola et al (2021) was used as a starting point, where the indicator consisted of blue and yellow areas. The blue areas disappeared when exposed to humidity above the threshold. This allowed development of smart tags following the principle of Upcode reading app as described in Figure 2.2.1. There, the blue bar at the bottom of the smart tag disappears when exposed to elevated humidity. The code contains different meta-data depending on the state of the functional bar, and UpCode reader (https://www.upcodeworld.com/) can detect this change in order to provide different information based on the state of the sensor.

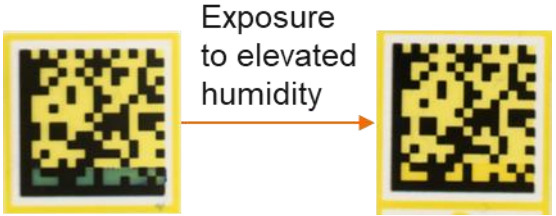


Figure 2.2.1 Humidity indicating smart tag principle for the Greek demo case.

In Figure 2.2.2 the full smart tag concept is illustrated. The transparent film containing the smart tag is attached tightly to the wood surface by using an adhesive that does not allow air leakage. When the smart tag is printed at the reverse side of the transparent film and is in contact with the wood, the humidity of the wood is monitored.

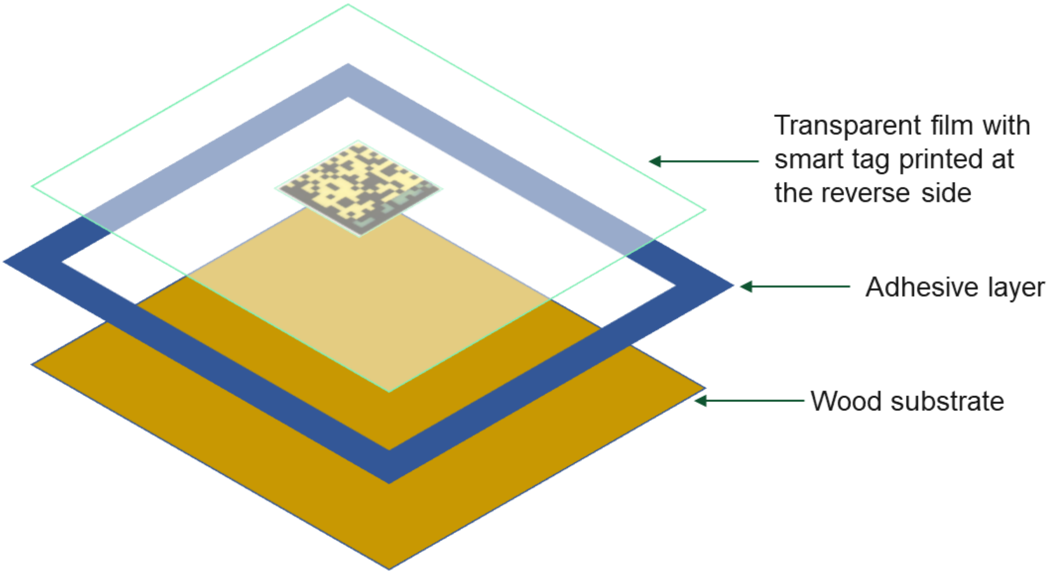


Figure 2.2.2 Smart tag concept for the Greek demo case.

For the Belgian demo case the development goals were to:

1) Test printing of smart tags on different textile substrates. Label substrates were used as a benchmark.
2) Attach the smart tags to textile substrates in a durable way.

A thermochromic ink was used as the temperature sensing area. The coloured area at the bottom of the code disappeared when exposed to a temperature above the threshold as illustrated in Figure 2.2.3.

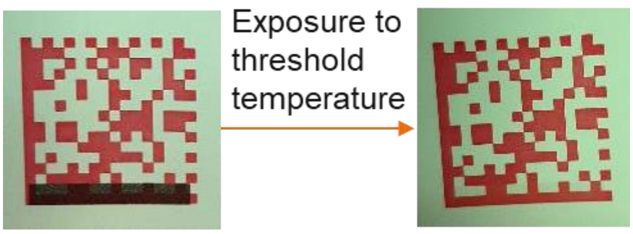


Figure 2.2.3 Temperature sensing smart tag principle for the Belgian demo case.

In Figure 2.2.4 the full smart tag concept is illustrated. The smart tags are directly printed on the textile substrate. To allow smart tag lifetime durability a protective transparent layer can be used as described in Hakola et al (2023).

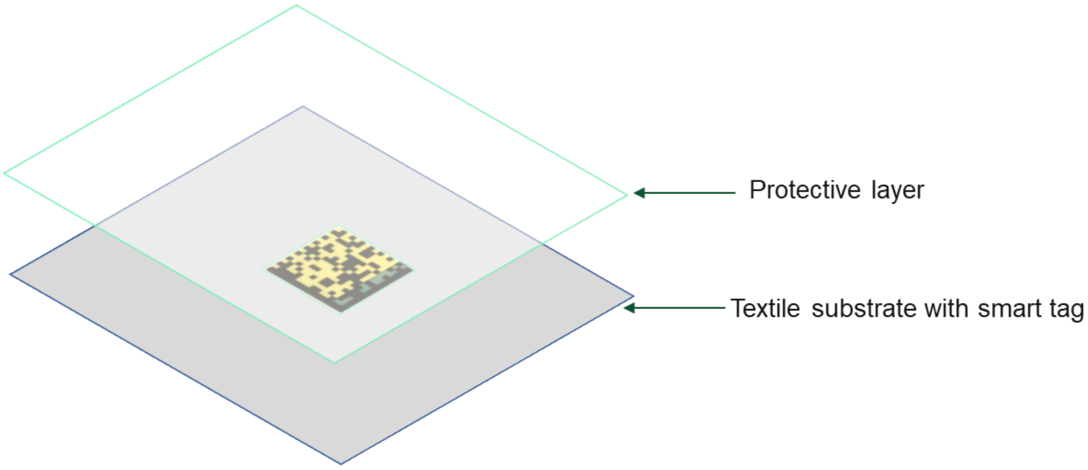


Figure 2.2.4 Smart tag concept for the Belgian demo case.

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3 Humidity indicator

3.1 Materials and methods

The following chemicals and reagents were used for development of the humidity indicator:

- Brom Cresol Green (BCG) pH colorant; visual transition interval 3.8-5.4 from yellow to blue (CAS 76-60-8)
- Calcium chloride $CaCl_2$ (CAS 10043-52-4)
- 1-methoxy-2-propanol (1ME2PRO) as the main solvent (CAS 107-98-2)
- PVP (Poly Vinyl Pyrrolidone) with molecular weight 10000 as a binder allowing adhesion on plastic substrates (CAS 9003-39-8)
- Surface active agent Dynol to adjust the surface tension of the ink into a proper level for inkjet printing (CAS 169117-72-0)
- Ammonia to adjust pH of the inks (CAS 7664-41-7)
- Lactic acid (CAS 50-21-5)

The following plastic substrates were selected:

- PET (Poly Ethylene Terephthalate)
- PP (Poly Propylene)

Inkjet printer with laboratory scale printheads was used for printing the indicators: DMP-2831 (Fujifilm Dimatix) with single-use 16 nozzle printheads and 10 pl drop size, printing resolution 1270 dpi.

Adhesives for attachment on wood were 3M 467MPF (3M) and barrier adhesive EL-92734-38 (Adhesives Research). Cover foils used to detect the moisture permeation through the cover foil were PET, Melinex ST506 (DuPontTeijinFilms) and barrier foil UBF-510 (3M).

3.2 Results

The results encompass a selection of detection chemistry, ink formulation to achieve compatibility on plastic substrates, and analysis of exposure to humidity.

3.2.1 Detection chemistry

The same detection chemistry as used in Hakola et al (2021) was selected, where the humidity indicator consisted of two inks:

1) Indicator ink including a moisture absorbing substance and an indicator dye,
2) Acid ink containing an organic non-volatile acid.

Moisture absorption into the system enables the diffusion and subsequent reaction of the inks resulting in a visual colour change as presented in Figure 3.2.1.1. The colour change is irreversible.

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Figure 3.2.1.1 Principle of the humidity indicator

3.2.2 Ink formulation and printing

The indicator ink has been previously formulated to be compatible with paper substrates. However, for the Greek demo case a transparent plastic substrate was required to be able to see the visual colour change through the substrate. Therefore, the solvent carrier of the inks had to be re-formulated to achieve sufficient adhesion and print quality on non-porous plastic substrates.

The final ink composition of 1) indicator ink consisted of:

- 4% PVP in 1ME2PRO (4g)
- BCG 0.16 wt-% in 1ME2PRO (10g)
- $CaCl_2$ (0.8g)
- 0.1 % of Dynol (10 mg)
- Ammonia to adjust the pH to 8.4 of the indicator ink (90 µl)

The final ink composition of 2) acid ink consisted of:

- 4% PVP in 1ME2PRO (4g)
- 0.1 % of Dynol (10 mg)
- 1 wt-% lactic acid

These formulations resulted in good inkjet printability and adhesion on plastic substrates PET and PP with one and two ink layers, respectively. On PET the blue area was clearly blue, but on PP somewhat greenish. The viscosity of the inks was 5.68 cP and surface tension 29.0 mN/m, thus being almost ideal for inkjet printing. The optimum values for inkjet printing are 8-12 cP and 24-36 mN/m, respectively.

3.2.3 Exposure to humidity

Humidity indicators were exposed to humidity standards 0%, 22%, 33%, 43% and 72% to analyze the threshold humidity where the indicator starts to react to elevated humidity i.e. colour change starts. Results after 4 and 96 hours of exposure are presented in Table 3.2.3.1.

Table 3.2.3.1 Exposure to different humidity levels after 4 and 96 hours of exposure on PET and PP substrates.

Humidity	0%	22%	33%	43%	72%
PET 0 h					

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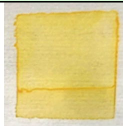
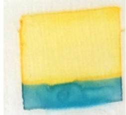
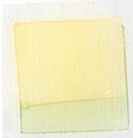
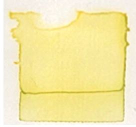
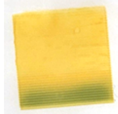
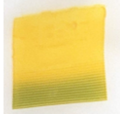
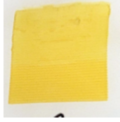
Humidity	0%	22%	33%	43%	72%
PET 4 h					
PET 96 h					
PP 0 h					
PP 4 h					
PP 96 h					

With the PET substrate the colour change starts for 33% and 43% humidity after 4 hours, but in a 72% humidity the change starts later. However, such a high humidity causes the whole printed area to start migrating on the substrate. At 22% humidity there is some colour change after 4 hours, but then the change does not progress. With the PP substrate the colour change is slower and the biggest colour change can be seen only after 4 hours. Also, with this substrate there is visual colour change already at 22% and at 72% the change starts faster than on PET. The results indicate that by substrate selection the speed of the colour change could be affected.

To analyze if the humidity indicator could be used for semi-quantitative analysis, the speed and intensity of the colour change was monitored. Here, the 22% humidity standard was not used since the change was not very clear in the previous test. Colour values were measured at each test point using CIELAB colour space that is intended to mimic the nonlinear response of the visual system. Colour difference ΔE* between the starting point and the test point were calculated with the formula:

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

L* is the lightness of the colour (0 is black, 100 is white), a* is the position between red and green (negative value for green, positive value for red), and b* is the position between yellow and blue (negative values for blue, positive values for yellow). The measurement was done for the blue bar, since that is the area whose colour change the mobile phone app is analyzing. The corresponding graphs for PET and PP are presented in Figures 3.2.3.1 and 3.2.3.2, respectively.

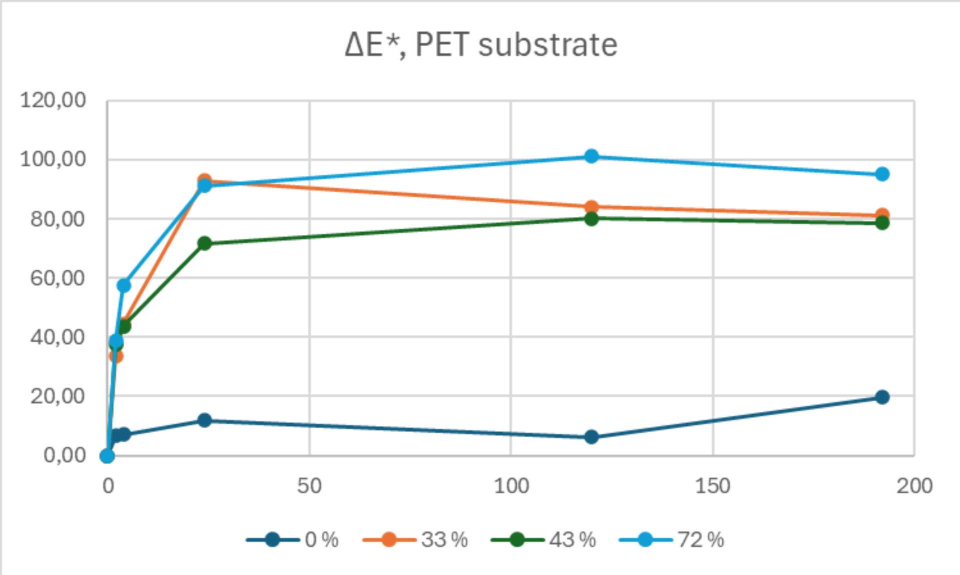


Figure 3.2.3.1 Colour difference on PET substrate at different humidity levels.

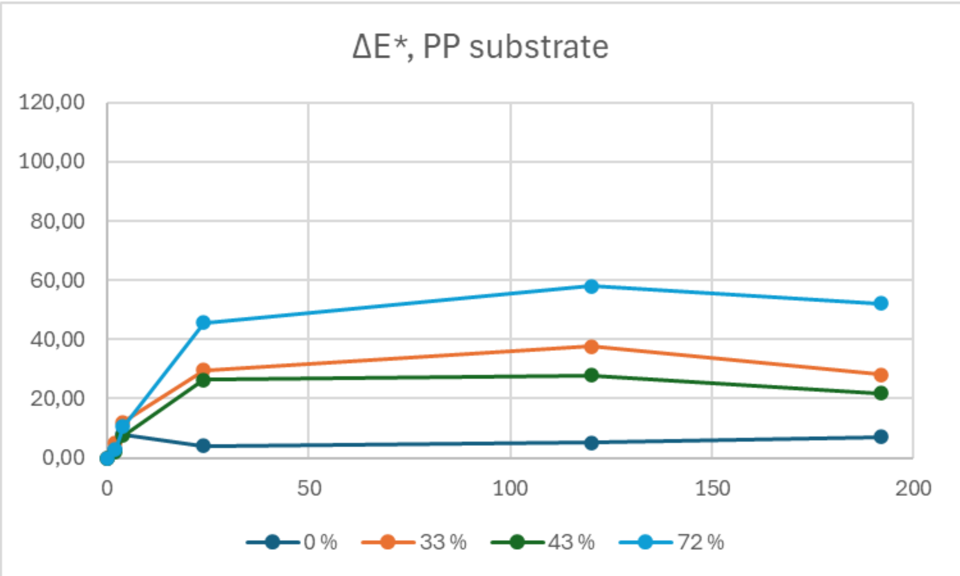


Figure 3.2.3.2 Colour difference on PP substrate at different humidity levels.

The biggest change in colour occurs during the first 24 hours, and is bigger for the PET substrate. After that the changes are smaller, and even decrease. This might be because some samples start to turn from yellow to green (see Table 3.2.3.1). Since CIELAB coordinates are based on measuring the actual colours (a* is red vs green, b* is yellow vs blue) the colour change first from blue to yellow and then to green might explain the decrease in colour difference ΔE after the first 24 hours. Yellow and blue are measured with b* value and green with a* value. These two variables behaving differently influence total value of ΔE. As an example, a* and b* for PET and PP substrate are presented in Figure 3.2.3.3 and 3.2.3.4, respectively. Therefore, the measurement values should be observed next to visual images. It should be noted that also at 0% there starts to occur some colour change, thus indicating that there might be some stability issues with the inks with longer exposure times.

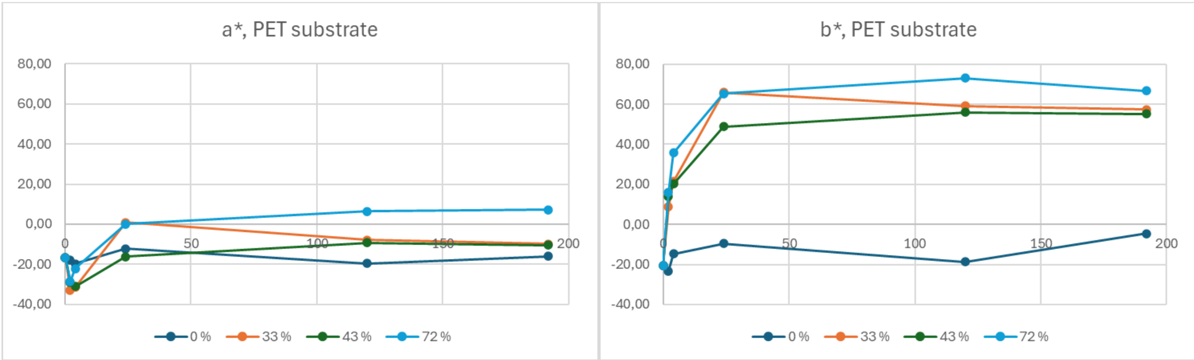


Figure 3.2.3.3 a* and b* values on PET substrate at different humidity levels.

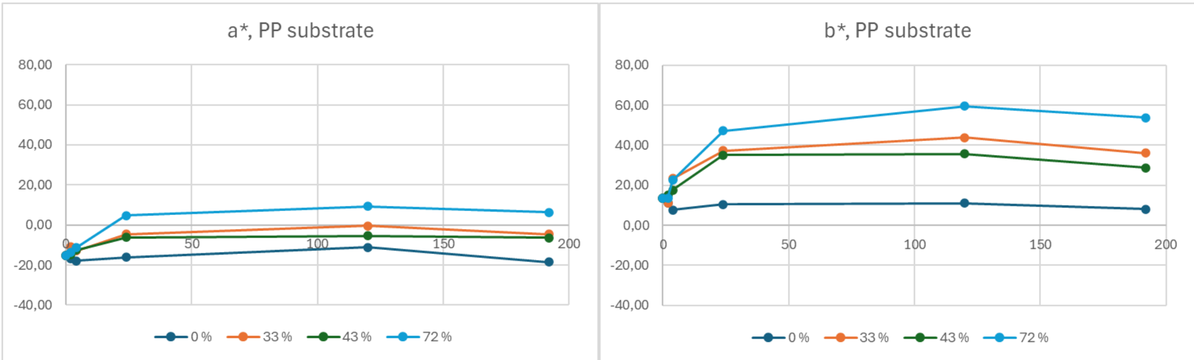


Figure 3.2.3.4 a* and b* values on PP substrate at different humidity levels.

The results do not give a definite answer if the developed humidity indicator could be used for semi-quantitative analysis after the first 24 hours, at least to analyze the duration of the exposure. However, it might be possible to analyze which humidity level the indicator has been exposed to, since the colour difference ΔE has a clearly different value per exposed humidity. In this case further studies would be needed to understand why the colour change is the smallest with 43%, since this wasn't the smallest humidity value used.

3.2.4 Attachment on wood

Attachment on wood and moisture permeation through cover foil was studied with laminated structures presented in Figure 3.2.4.1. Commercial humidity indicators were laminated between PET foils and between barrier foil having WVTR $5x10^{-4}$ $g/m^2/day$, using conventional transfer adhesive 467MPF and barrier adhesive EL-92734-38 with 30 mm width. Structure a) was used to detect the moisture permeation through foils and adhesives. Structure b) was used to compare the adhesion strength of the adhesives to the wood surface. The samples were exposed to 21°C, 60%rH conditions in a weathering chamber.

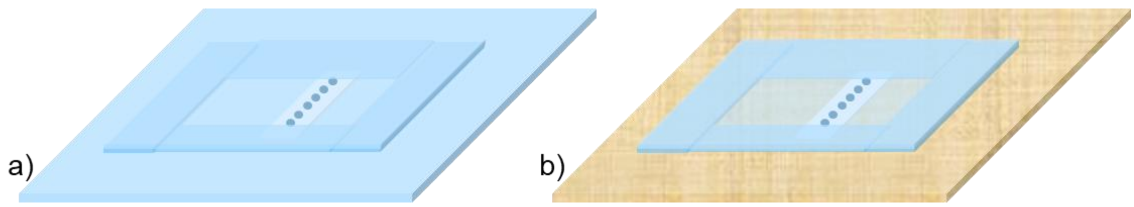


Figure 3.2.4.1 Commercial humidity indicators laminated between a) foils, b) foil and birch veneer.

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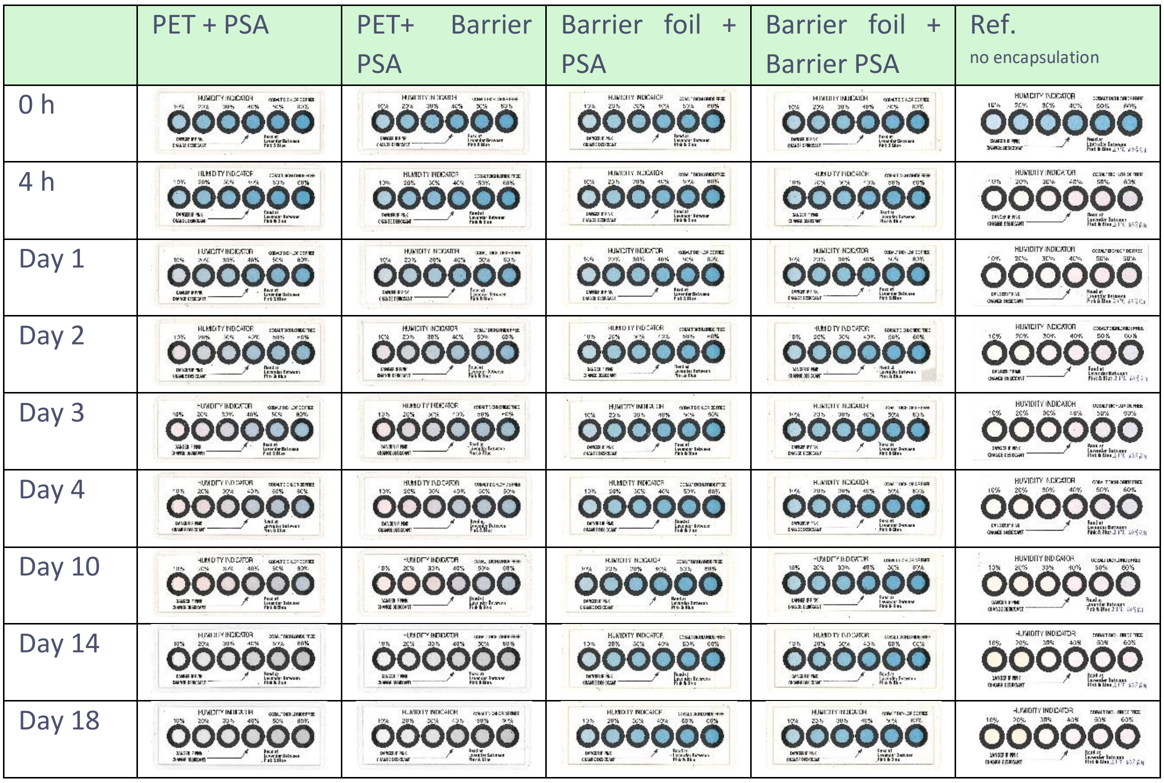

Figure 3.2.4.2 Weathering of commercial humidity indicators laminated between foils under 21°C, 60%rH.

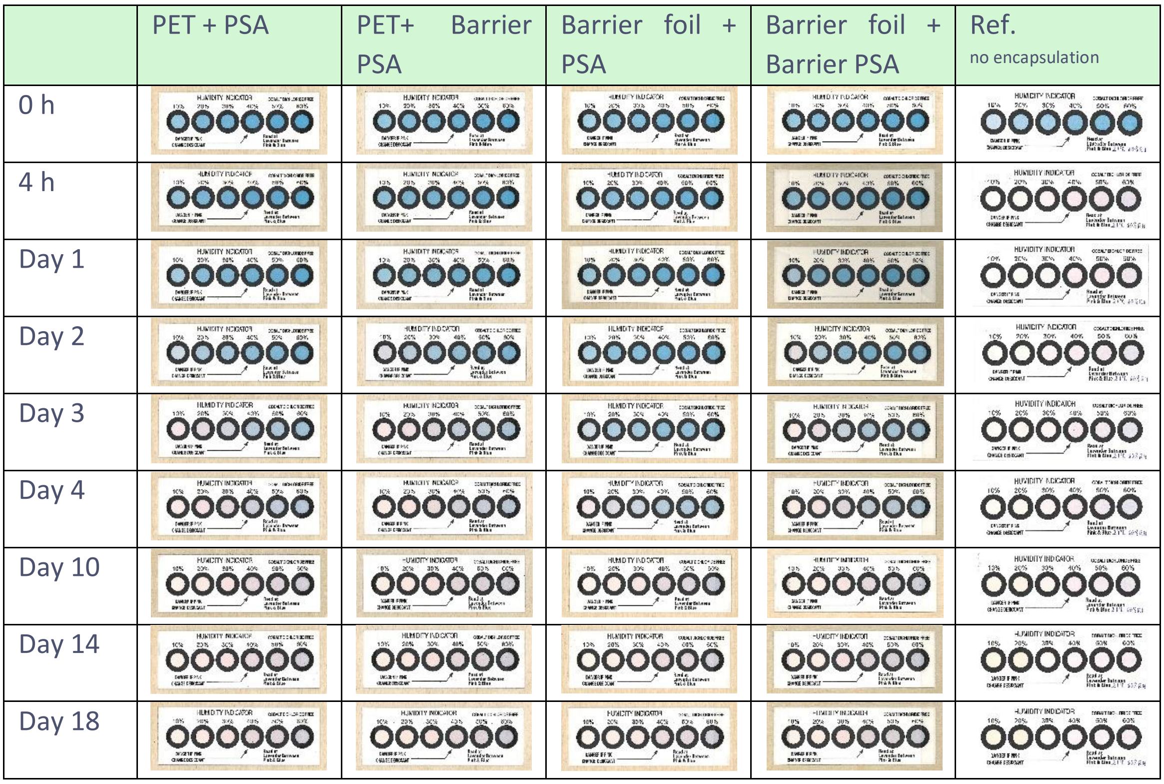

Figure 3.2.4.3 Weathering of commercial humidity indicators laminated between foils and birch veneer under 21°C, 60%rH.

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Figure 3.2.4.2 shows that the use of barrier foil and barrier adhesive is slowing down the moisture permeation in laminated structure. Humidity indicators laminated with PET foil started to react after two days exposure whereas indicators laminated with barrier materials stayed unchanged for 18 days.

Adhesives are playing a bigger role when the indicators are laminated on wood surface. Conventional adhesive, 467MPF, has better adhesion on the veneer surface resulting slower moisture permeation in both structures, with PET and barrier foil.
Barrier foil slows down the moisture permeation trough the foil, however moisture is penetrating though veneer and adhesive. The barrier foil used has a high quality barrier, with a WVTR of only 5x10-4 g/m^2/day (Water Vapour Transition Rate). Using that expensive material is probably not reasonable, but using some less expensive material could be considered if moisture permeation through PET needs to be slowed down.

In the second weathering test, humidity indicators processed on PP substate were laminated on 4 mm thick veneers using a 467MPF adhesive together with PET foil and barrier foil. Two types of structures, presented in Figure 3.2.4.4, were prepared. The adhesive width around the humidity indicator was 30 mm. In structure a) the printed humidity indicator was placed facing the veneer surface detecting the humidity absorbed to the wood. In structure b) the humidity indicator side was facing the cover foil, indicating the moisture permeation through the foils. The samples were exposed to 21°C, 60%rH conditions in a weathering chamber.

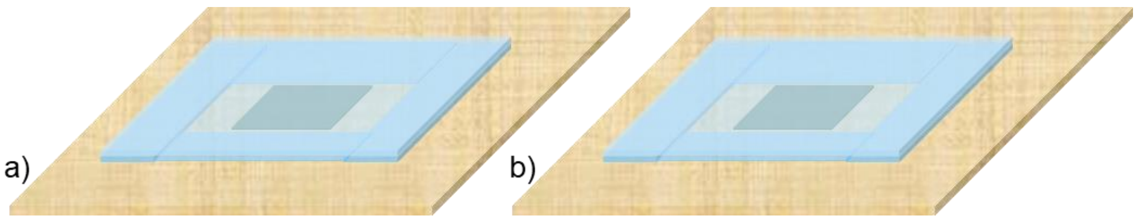
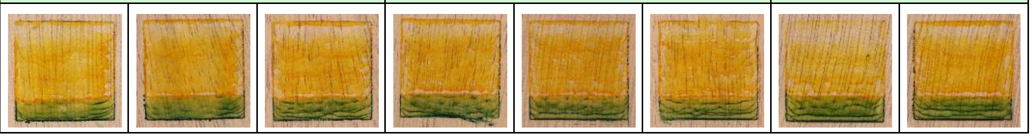
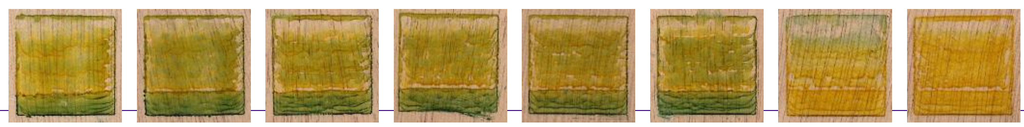


Figure 3.2.4.4 humidity indicators laminated between foil and 4 mm veneer a) indicator side facing the veneer b) indicator side facing the top foil.

Color changes in humidity indicators facing the wood surface were observed already in the first measurement point after a 44-hour exposure. Pictures of the humidity indicator during exposure are presented in Figure 3.2.4.5. When the humidity indicators are facing the cover foil, no color change was observed during a 360 hours exposure, Figure 3.2.4.6. It can be concluded that both PET and barrier foils are preventing well moisture permeation, and leakage through the adhesive layer is not occurring either. These results show that, when the printed humidity indicator side is facing the wood surface, it is indicating the humidity changes in the wood. Cover foil moisture barrier properties didn't play a significant role in the two-week period studied.

	PET	Barrier	Ref. no encapsulation
0 h			



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44 h								
95 h								
210 h								
360 h								

Figure 3.2.4.5 Weathering under 21°C, 60%rH, humidity indicator facing the veneer surface.

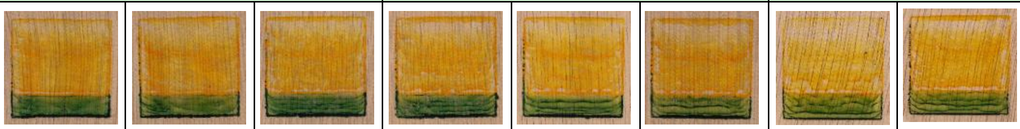
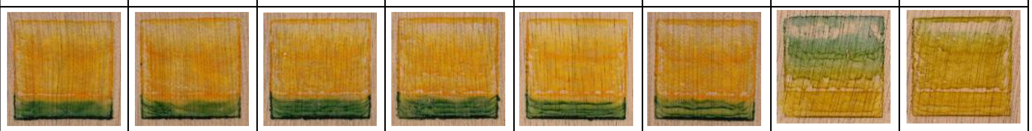
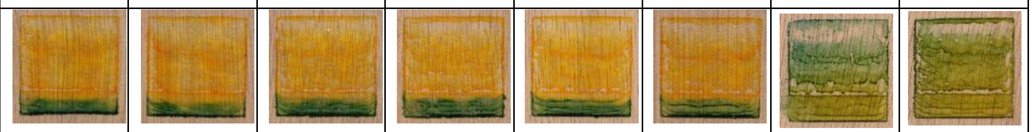
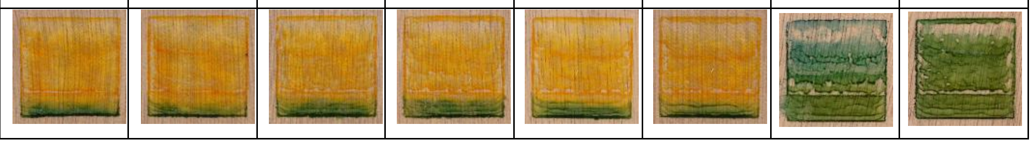
	PET			Barrier foil			Ref. no encapsulation	
0 h								
44 h								
95 h								
210 h								
360 h								

Figure 3.2.4.6 Weathering under 21°C, 60%rH, humidity indicator facing the cover foil.

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4 Temperature indicator

4.1 Materials and methods

The following substrate materials were used for printing the smart tags:

- Label materials
    - PET Melinex ST506 (PET)
    - PET White RR28 (TC50-RR28_HD70) (PET white RR28)
    - PET White RC10 (TC50-RC10_HD70) (PET white RC10)
    - PP White (FTC60/RP37) (PP white)
    - PE RI-837/85 PE Gloss Clear TC8 AP901 W G 62 (PE)
    - PE Matt white (FTC85/RP37) (PET matt white)
    - PE carrier (PE carrier)
- Textile materials
    - 100% Cellulose with latex, from VTT (cellulose 100)
    - 67% cellulose with 33 % viscose, 31 g/m^2, from VTT (cellulose 67)
    - 33% cellulose with 67 % viscose, 52 g/m^2, from VTT (cellulose 33)
    - 10% cellulose with 90% viscose, 68 g/m^2, from VTT (cellulose 10)
    - Recycled PET matt side, from Belgian demo case (Recycled PET matt)
    - Recycled PET glossy side, from Belgian demo case (Recycled PET glossy)
    - Recycled PET textile, from Belgian demo case (Recycled PET textile)

The smart tags were printed with two inks:

- Graphic ink
    - Sun Chemicals 049-37329: Special Research Magenta:FJ49
- Thermochromic ink
    - LCRHallcrest Black thermochromic ink – Therm. w/b scr. ink – Activation temperature: 31°C

2D barcodes were printed with flexography using RK Flexiproof 100 onto the selected substrates and using the graphic ink. The cell volume of the anilox rollers was either 5 ml/m^2 or 9.9 ml/m^2 depending on the porosity of the substrate. The printing speed was 10 m/min and the printed layer was dried in the convection oven at 50-80°C for 5 min.

The thermochromic layer was printed on the top of the 2D code using a manual screen printing. The thermochromic layer covered the bottom rows of the printed 2D codes. The thermochromic ink layer turned from black to transparent above the activation temperature. Above the activation temperature, the code directs to a different webpage than below the activation temperature. The mesh count of the screen was 325 lines/inch and the ink layers were dried at 80°C for 5 min.

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4.2 Results

4.2.1 Printing

Codes of different sizes were printed, starting from 1 cm^2 to 4 cm^2. 2D barcodes and thermochromic ink layers were successfully printed onto the selected plastic substrates. Some thermochromic layers were printed twice to ensure good quality of the code elements and to create enough dark layer to cover the code evenly. This was done in particular with textile substrates. During the printing of the thermochromic layer, some layer-to-layer registration issues were seen due to the substrate shrinking and wrinkling during the drying of the 2D code layer. The most shrinking was seen with PE substrates. Some textile substrates suffered from poor ink transfer, and uneven ink sinking into the porous structure of the substrate. As a result, print quality differences were seen between parallel sheets and also within the single printed sheet. In addition, the code reading was not reliable with smaller codes or the reading distance needed to be increased.

Thermochromic layers changed their colour from black to transparent above the activation temperature and also returned to their colored state once the samples were returned to RT, as shown in Figure 4.2.1.1. The 2D barcodes could be read at both stages (colored vs. transparent). On some textile substrates, the codes could not be read, although good colour change was seen. The printed code patterns sunk into the pores and irregularities of the surface, thus creating an uneven contrast and interfering with the reading. In particular, small codes with smaller reading distance encountered more severe reading issues.

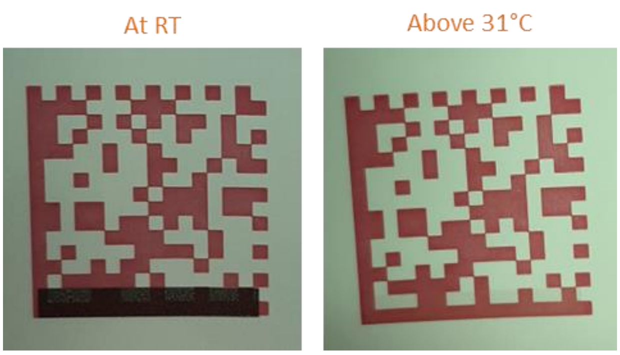

Figure 4.2.1.1 Change in color of the thermochromic layer from black to transparent as the temperature increased above the activation temperature of 31°C.

4.2.2 Analysis of print quality

The width and height of smart tag cells were measured from 2 cm^2 codes and imaged with a microscope to analyze the print quality on different substrates. An ideal cell width and height would be 1.25 mm. The results are summarized in Figure 4.2.2.1. Since the cells have bigger dimensions on textile substrates than on label substrates, there is more ink spreading on the more porous substrate types i.e. the textile ones. On Recycled PET textile substrates, the visible surface texture affects the print quality and makes the cells smaller. The smart tags were not readable with a mobile phone on this substrate, as was the case also with Cellulose 67 substrates. There were also reading challenges with the other textile substrates, again, due to visibility of the surface structure. Specifically on the cellulose substrates, the smart tags were not dark enough to be read reliably, since the ink had penetrated into the porous substrate structure.

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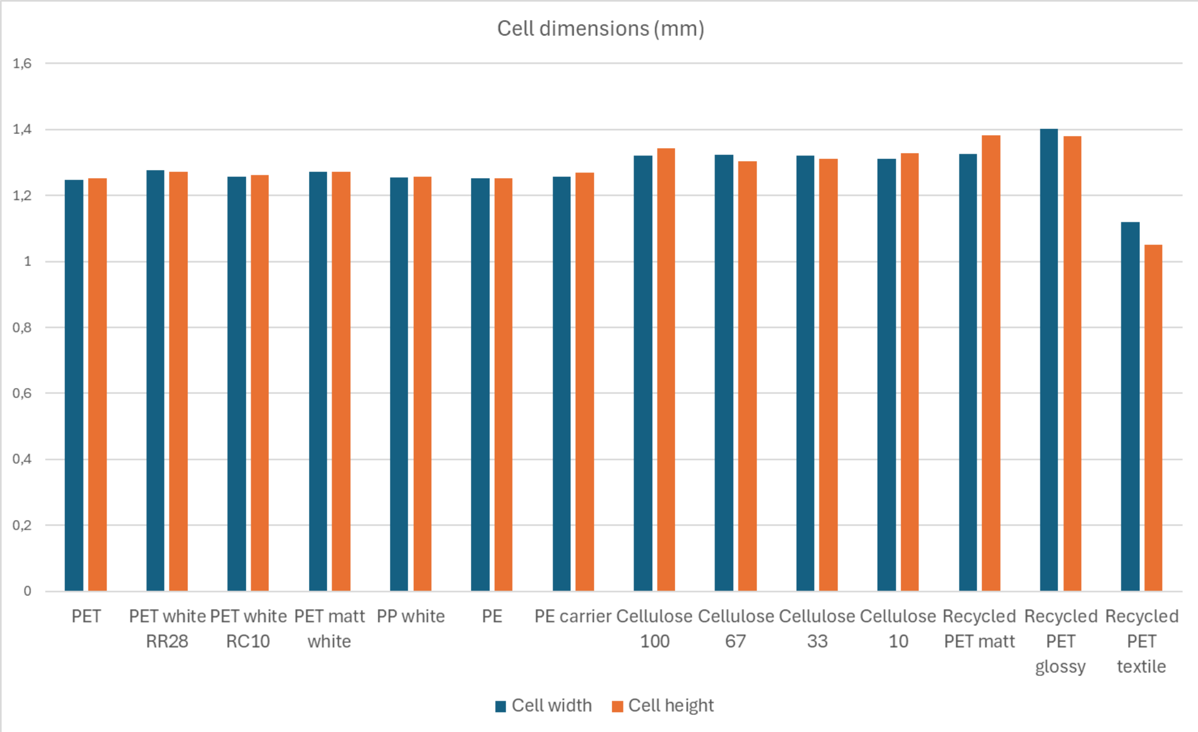
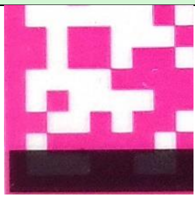
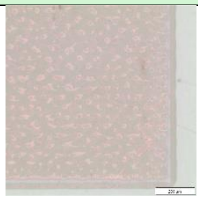
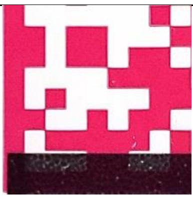
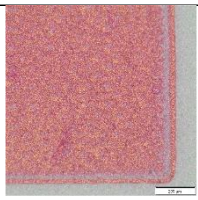


Figure 4.2.2.1 Cell width and height on different substrates

4.2.3 Exposure to threshold temperature

Table 4.2.3.1 shows the smart tags in room temperature (+23°C) and in +40°C, and microscopic images of the tag. The magnifications are 2x (image width 1 cm) and 5x (image width 0.1 cm). The colour change of the thermochromic bar is clearly visible on all substrates. With the textile substrates the surface texture can be seen on the microscopic images and how it has affected the printing quality compared to label substrates. This is the most likely reason why reading smart tags on textile substrates was challenging.

Table 4.2.3.1 Microscopic images and behaviour of smart tags in room and +40°C temperature on different substrates

Substrate	Microscope magnif. 2x	Microscope magnif. 5x	Room temperature	+40°C
PET				
PET white RR28				

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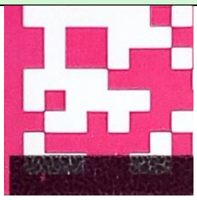
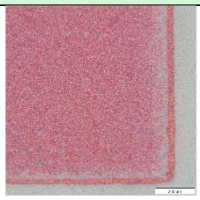
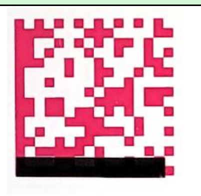
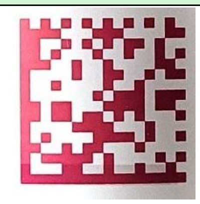
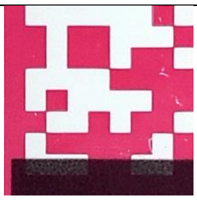
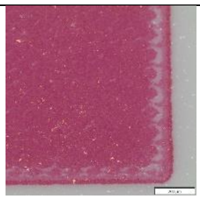
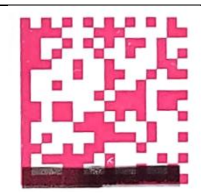
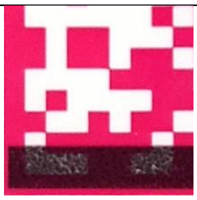
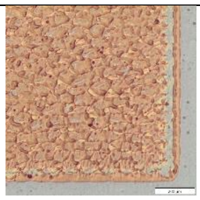
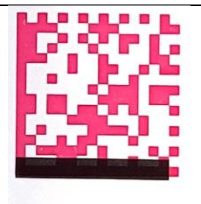
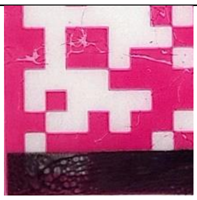
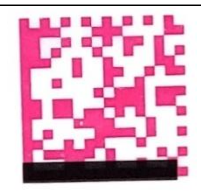
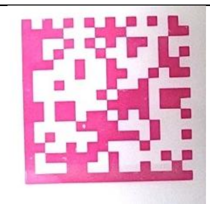
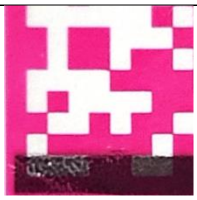
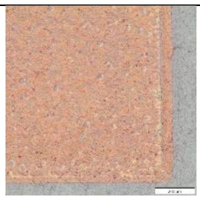
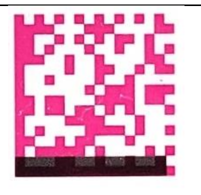
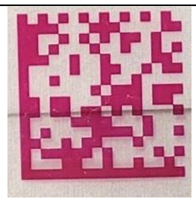
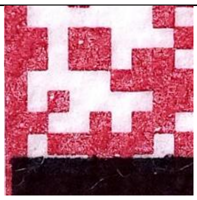
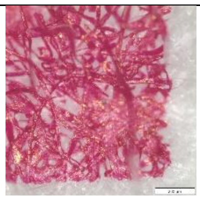
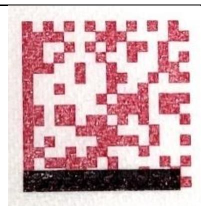
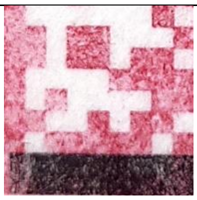
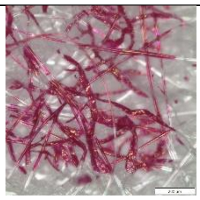
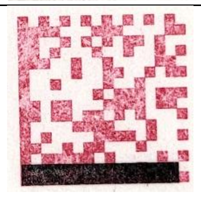
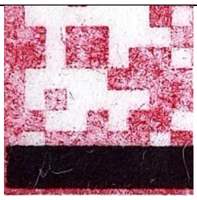
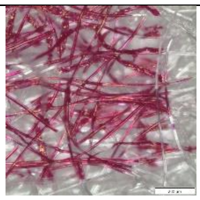
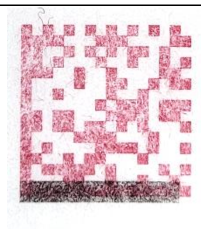
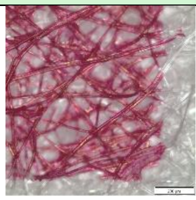
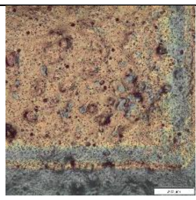
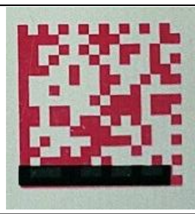
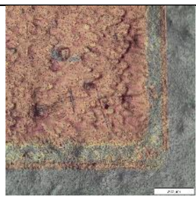
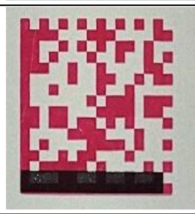
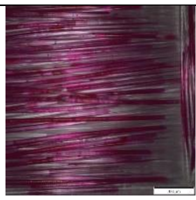
Substrate	Microscope magnif. 2x	Microscope magnif. 5x	Room temperature	+40°C
PET white RC10				
PET matt white				
PP white				
PE				
PE carrier				
Cellulose 100				
Cellulose 67				
Cellulose 33				

Substrate	Microscope magnif. 2x	Microscope magnif. 5x	Room temperature	+40°C
Cellulose 10				
Recycled PET matt				
Recycled PET glossy				
Recycled PET textile				

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5 Conclusions

This report has summarized the smart tag concepts developed for selected DigInTraCE project demo cases. It concludes that both humidity and temperature sensors are available for integration into the demo cases in *WP6*. Fine-tuning of the technologies can be done to meet the final demo case requirements, e.g. targeting a specific reaction speed and/or sensitivity.

The Greek demo case has pointed that a formaldehyde indicator would also be of interest due to special requirements of their demo case, as reported in WPs 5 and 6. Since a suitable chemistry from previous activities exists at VTT, the experimental part in M19-M36 will focus on analyzing the feasibility of tailoring a printable formaldehyde sensor (to be integrated with a data carrier) into a smart tag concept. The Belgian demo is still considering how to use the smart tags in their demo case.

The next steps will also focus on data integration and integrity by investigating how to integrate decentralized, GAIA-X compliant data management and storage into the physical tags. Furthermore, compatibility of the smart tag technology with existing data carrier standards, such as ISO/IEC 15459:2015 (Information technology — Automatic identification and data capture techniques — Unique identification) and GS1 Digital Link, will be analysed.

To investigate stakeholder perception, particularly from the industry towards smart tags, targeted interviews will be implemented with selected key stakeholders outside the project consortium during M24-M36. The stakeholders might also represent industrial sectors outside the project demo cases. The emphasis will be given to technology developers related to smart tags, end users of circular products, and end-of-life operators. For this purpose, an interview structure will be designed to include the most relevant discussion points that have arisen during the project implementation. If feasible, an additional workshop will be organized as a side-event for a suitable event focusing on circular economy.

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6 References

Ghaani, M; Cozzolino, C.A.; Castelli, G; Farris, S. (2016) *An overview of the intelligent packaging technologies in the food sector*. Trends in Food Science & Technology, Volume 51, pp 1-11.

Gligoric, N; Krco, S; Hakola, L; Vehmas, K; De, S; Moessner, K; Jansson, K; Polenz, I; van Kranenburg, R. (2019) *SmartTags: IoT Product Passport for Circular Econo-my Based on Printed Sensors and Unique Item-Level Identifiers*. Sensors, vol. 19, no. 586, 26 p.

Hakola, L; Vehmas, K; Smolander, M. (2021) *Functional Inks and Indicators for Smart Tag Based Intelligent Packaging Applications*. JAPR Journal of Applied Packaging Research, Vol. 13, No. 2

Hakola, L; Jansson, E; Palola, S; Tenhunen-Lunkka, A; Lahtinen, J. (2023) *Durable and sustainable smart tags for identity management and condition monitoring: case study for reusable packaging*. Packaging Technology and Science.

Sipiläinen-Malm, T; Hurme, E. (2008). *Printed oxygen indicators for food packages.* In European Coatings Conference: Food Contact Coatings. Berlin, Germany.

Tenhunen-Lunkka, A; Lahtinen J; Hakola, L; et al. (2023). *Operational environment review for reusable packaging used in fast moving consumer goods: 4everPack.* VTT Technical Research Centre of Finland VTT Technology No 415, 2023. doi:10.32040/2242-122X.2023.T415