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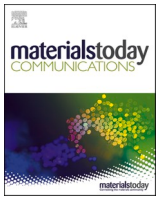

Characteristics of secondary fibres derived from recycling processes of fibreboard manufacturing residues and post-consumer wood waste

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ABSTRACT

Recycling of post-consumer fibreboards has gained significant traction in both academic research and industrial applications. The quality of secondary fibres plays a crucial role when reusing them in fibreboard production. This study presents a comprehensive analysis of recycled (hereafter referred as secondary) fibres recovered through modified thermo-mechanical pulping and steam treatment methods, in comparison to virgin fibres. The fibres were systematically characterized in terms of their physical, chemical, and morphological properties, supplemented by microscopic analysis. Results showed that the average fibre length of steam-treated secondary fibres derived from post-consumer fibreboards and fibreboard production (processing) residues was comparable to that of virgin fibres, whereas secondary fibres from solid wood waste were 18 % shorter due to identified presence of hardwood species. All secondary fibre types exhibited higher fines content, increased surface roughness, and elevated formaldehyde emissions relative to virgin fibres. FTIR spectroscopy and elemental nitrogen analysis, confirmed the presence of residual resin, which contributes to a higher pH and increased alkaline buffering capacity in secondary fibres. Dynamic vapour sorption (DVS) analysis revealed reduced water uptake and increased hysteresis in secondary fibres from steam treatment. Scanning electron microscopy (SEM) and inductively coupled plasma (ICP) analysis identified inorganiccontaminants and elevated concentrations of elements—specifically sodium, aluminium, and lead, particularly in fibres from post-consumer fibreboard fractions. Finally, Fibre blends containing 15 % or 25 % (w/w) secondary fibres from fibreboard processing residues exhibited comparable properties to virgin fibre benchmarks. This study establishes a fundamental property baseline for secondary fibres, providing a framework for the formulation and optimization of future sustainable composites.

1. Introduction

The valorization of bio-residues is a fundamental pillar of the global transition toward a circular bioeconomy. While previous studies have extensively investigated the utilization of bast and agricultural fibre for applications ranging from sustainable textile production to rural infrastructure development [1,2], the industrial-scale recycling of wood waste is equally critical for achieving resource circularity within the wood-based panel industry [3]. Within this framework, the wood composite industry plays a pivotal role, particularly through the production of fiberboard [4]. Fibreboard is a wood composite widely used in furniture and interior applications due to its low cost and high machinability, manufactured by hot pressing of wood fibres glued with resins and wax additives [5,6]. Fibreboard is mainly manufactured from softwood fibres like Norway spruce (*Picea abies* (L.) H. Karst.) and Scots pine (*Pinus sylvestris* L.), while hardwoods like beech (*Fagus sylvatica* L.) and oak (*Quercus* spp) are used less often [7,8]. Virgin fibres for fibreboard are primarily produced via the thermo-mechanical pulping (TMP) process, which is cost-effective and well-established [9,10]. Amongst the fibreboard types, medium-density fibreboard (MDF) dominates the market, with steadily increasing global production. However, its short service life (10,7–12.1 years) has led to a growing volume of post-consumer waste [10–12]. Recycling post-consumer MDF and wood waste promotes the efficient utilization of wood resources and aligns with the cascading use principle. Increasing the incorporation of these recycled materials enhances the sustainability and circularity of wood-based panels while simultaneously contributing to climate change mitigation [13,14].

Recycling fibreboard waste through mechanical, thermo-mechanical, or chemo-thermo-mechanical methods, however, remains

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challenging due to process complexity, effluent management, and, most critically, deteriorated fibre quality, whether from fibreboard production (processing) residues or post-consumer fibreboards [9,15,16]. Despite these challenges, recycling fibreboard is environmentally preferable to landfill or incineration [17,18]. Reusing wood waste in fibreboard manufacturing reduces the demand for virgin wood, enhances carbon sequestration, and lowers greenhouse gas emissions [19]. Therefore, establishing effective fibreboard recycling enhances the move towards more sustainable wood-based panel production [20,21]. While recycled solid wood is increasingly used in particleboard (PB) and fibreboard, post-consumer fibreboard presents greater challenges due to the presence of hazardous chemicals in adhesives, coatings, and fire retardants [14]. Research has explored the recycling of fibreboard waste, such as for making PB or partial reuse in new fibreboards [4,22].

The performance of fibreboard depends on fibre properties, adhesive characteristics, and the manufacturing process [23–25]. Recycled fibres commonly exhibit worsened properties [6,26]. For example, secondary fibres from fibreboard processing residues can be 30 % shorter, with higher pH and buffering capacity [27]. Fibre damage and contamination are the key limitations [15,28,29]. This has driven research into resin removal for better preserving fibre integrity [29–31]. Urea-formaldehyde (UF) and melamine-urea-formaldehyde (MUF) dominate the market as adhesives in European PB and MDF production, with phenolic and isocyanate resins used in smaller volumes (2–3 %) [32]. Acid-based recycling removes the most UF resin at temperatures below 100 °C, compared to alkaline solutions and water [29]. Alternatively, Hydrothermal approaches—steam treatment, steam refining, and steam explosion—also break down resin and release fibres for reuse [6,33,34]. These can remove up to 85 % of UF resin, although often with fibre shortening and yield loss [34,35]. Steam treatment can also darken fibres (deep brown colour), due to the formation of chromophoric groups in the lignin following condensation and oxidation reactions [6,36].

Fibre characteristics, such as fibre length distribution, acidity, and morphology, strongly affect the performance of fibreboard [25]. Wood fibres are inherently variable across tree species, geographic origin, processing conditions, and even by location within the same tree [7,37]. Recycled fibres typically contain more fines, irregular fibre bundles, and residual resin/wax, leading to altered chemical and physical properties compared to virgin fibres [31,35,38]. This often reduces fibreboard performance, with shorter fibres, contamination, and resin residues impairing strength and durability [6,39]. Secondary fibres also exhibit reduced hemicellulose content, acidity, and increased buffering capacity, altering resin curing and gel time [30,40]. Fibreboard's internal bond strength depends on multiple factors, such as the manufacturing parameters (temperature, pressure, time), and resin type, content, and distribution [23,24]. The fibres' acidity strongly affects the internal bond strength of MDF with UF resins. [25]. These factors weaken inter-fibre bonding and affect water resistance and formaldehyde emissions [8,35,41,42]. The quality of recycled fibres varies with recycling methods and waste source, limiting the incorporation ratio without significantly compromising panel properties [7,41,43].

Although studies have examined both post-consumer waste and processing residues, there are limited studies on a comprehensive analysis of recycled fibres obtained from those waste fractions being available at different locations, the industry or the wood waste yard [28,29,35]. To advance circular use of fibreboard, it is essential to systematically characterize secondary fibres from various sources and recycling methods and link their properties to fibreboard performance.

This paper investigates recycled fibres obtained from processing residues and post-consumer wood (including fibreboards and solid wood) using modified thermo-mechanical pulping (mTMP) and steam treatment (ST). Fibre properties—including particle size, water resistance, nitrogen content, chemical contaminants, pH, buffering capacity, and formaldehyde emissions—were systematically analysed to assess their potential in new fibreboard production.

2. Materials and methods

2.1. Materials

Virgin TMP fibres (Scots pine, Pinus sylvestris L.) and secondary fibres from mTMP process were obtained from IHD (Institut fuer Holztechnologie Dresden gGmbH, Germany). Secondary ST fibres were received from Dieffenbacher Gmbh Maschinen- und Anlagenbau (Eppingen, Germany). Secondary fibres were recovered from three distinct waste streams: virgin solid wood waste, fibreboard processing residues, and post-consumer fibreboards at their end-of-life (EOL). While the solid wood fractions were binder-free, the fibreboard processing residues sourced from Homanit GmbH (Losheim, Germany) contained urea-formaldehyde (UF) resin loadings ranging from 10 % to 14 % (w/w) relative to the oven-dry fibre mass. The binder type and content in the post-consumer fibreboards remain undetermined, as these materials were sourced directly from municipal waste disposal sites in France.

2.2. Recycled fibre sources

Fig. 1 shows the materials from the process of wood waste sorting for obtaining solid wood and fibreboard fractions. Veolia S.A. (Paris, France) collected and crushed post-consumer wood, which is then screened using a Dieffenbacher ClassiScreen (a roller screen) into three fractions: Fines (<18 mm), Macro (18–100 mm), and Oversize (>100 mm), with foils and light materials removed.

The target 'Macro' fraction is cleaned of impurities (e.g., stones, metals, hard plastics) using a TOMRA (details of company) X-ray sorter and then sorted into 'solid wood' and 'rest wood' fractions (purity >95 %) by TOMRA GAINnext optical sorters. After, the 'rest wood' fraction was further separated by combining Near-Infrared Spectrometer (NIR) technology to sort out the 'fibreboard' fraction from the residual fractions (e.g., particleboard, plywood, oriented strand board). Fibreboard processing residues (non-sanded, 3.3 mm thick, 850 kg/m^3) was and consists of ~80 % Scots pine (*Pinus sylvestris* L.) and ~20 % Norway spruce (*Picea abies* L.).

2.3. Fibre preparation

Fibre extraction was performed via three distinct pathways based on the raw material source and secondary fibre rates. Virgin wood, fibreboard processing waste, and sorted waste fractions (solid wood waste and post-consumer fibreboard) were initially processed by IHD using a drum chipper, then sieved to chip sizes between 3×3 mm^2 and 25×25 mm^2.

Standard TMP: Virgin TMP fibres were obtained through defibration at a steam pressure of 10.4 bar, a chip pre-heating time of 3–4 min in the digester, and a grinding gap of 40 μm.

Moisture modified TMP (mTMP): To address the typically low moisture content (MC) (~10 %), a main disadvantage of waste wood, the mTMP process utilized a patented, optimized digester configuration to enhance steam penetration into the chips [44]. To obtain the secondary mTMP fibres, the waste chips were blended with virgin wood chips based on dry mass at ratios of 15:85 and 25:75, respectively. Afterwards, the mixed chips were defibrated with the same settings as for virgin fibres. 100 % secondary fibres from solid wood waste were obtained with a steam pressure of 6 bar, a chip pre-heating time of 3–4 min in the digester, and a grinding gap of 80 μm.

Steam Treatment (ST): The post-consumer fibreboard fraction and fibreboard processing waste were processed by Dieffenbacher to produce chips ranging from approximately 5×5 mm^2 to 60×60 mm^2. These chips were fed directly into a pressure vessel without any pretreatment. Steam was then introduced into the vessel gradually (3–20 bar) under continuous agitation to ensure uniform steam exposure. The chips were subsequently defibrated into individual fibres

Fig. 1. Materials from the wood waste sorting process. (a) post-consumer wood mixture, (b) crushed, (c) sorted solid wood waste, (d) sorted post-consumer fibreboard fraction.

during controlled pressure release rather than a steam explosion process. Once the pressure returned to ambient (0 bar), the 100 % ST fibres were discharged with the help of the agitator.

All resulting fibres were then dried to a moisture content of approximately 6 % in either a flash dryer or pipe dryer. Table 1 provides an overview of secondary fibres, and their composition and source. Fig. 2 highlights images of the prepared fibres. The performance characteristics of MDF incorporating these specific secondary fibres are provided in our previous work [39].

2.4. Fibre size distribution

Fibre length distribution was measured by dynamic image analysis (QICPIC, Sympatec GmbH, Clausthal-Zellerfeld, Germany), and a laboratory ultrasonic sifter (VU200, Imal S.r.l., Italy) was used to determine the fibre size frequency based on mass ratio. For fibre characterization, wood fibres were manually pre-separated because fibres tend to tangle and agglomerate. They were then introduced using the QICPIC analyser's fibre feeder VIBROS and further separated using RODOS. A high-speed camera then measured the sieved fibre sizes (measuring range M9: 17 – 33,792 µm; 75 frames/second). The WINDOX software (Sympatec, GmbH, Clausthal-Zellerfeld, Germany) was used to determine fibre length (Feret max) and diameter (Feret min) from projected 2D contour images and to calculate size distributions [45,46]. The fibre size was characterized concerning the median (X_{50}) and mean based on a perfect cylindric volume model (Q_3). Five replicates of 1 g fibre were measured for the prepared fibres (Table 1). As a complementary measurement to the optical method, the fibres' geometric parameters were also characterized by sieving based on mass per fraction. Fibres underwent a stack of sieves comprised of sieve sizes 0.098 mm, 0.131 mm, 0.217 mm, 0.514 mm, 1.24 mm, and 1.98 mm. The sieving time was 3 min per batch.

Table 1
Overview of prepared secondary fibres (SW denotes solid wood waste, FW denotes fibreboard processing residues, PW denotes post-consumer fibreboard fraction, and "1" indicates fibres obtained from steam treatment, denoted as ST).

Abbreviation	Fibre source and secondary fibre rate (based on mass)
TMP: Virgin (V)	TMP fibre, 100 % virgin pine
V + 15 %SW	mTMP fibre, 15 % derived from solid wood waste
V + 25 %SW	mTMP fibre, 25 % derived from solid wood waste
100 %SW	mTMP fibre, 100 % derived from solid wood waste
V + 15 %FW	mTMP fibre, 15 % derived from MDF processing waste
V + 25 %FW	mTMP fibre, 25 % derived from MDF processing waste
V + 25 %PW	mTMP fibre, 25 % derived from post-consumer fibreboard
100 %FW1	ST fibre, 100 % MDF processing waste
100 %PW1	ST fibre, 100 % post-consumer fibreboard

2.5. Fibre morphological characteristics

A microscopic analysis of the virgin and secondary fibres was conducted using scanning electron microscopy (SEM). For SEM, the fibres were mounted on stubs using carbon tabs and coated with gold using an Emitech K550X sputter coater (K550X, Emitech Ltd., Kent, UK). Observations were made using a Philips SEM (XL 30, Philips, Eindhoven, Netherlands) operated at 10 kV accelerating voltage with images recorded digitally.

2.6. Dynamic vapor sorption (DVS)

DVS analysis was performed on wood powder (< 0.315 mm) prepared from the fibres using a DVS-ET-VID (Surface Measurement

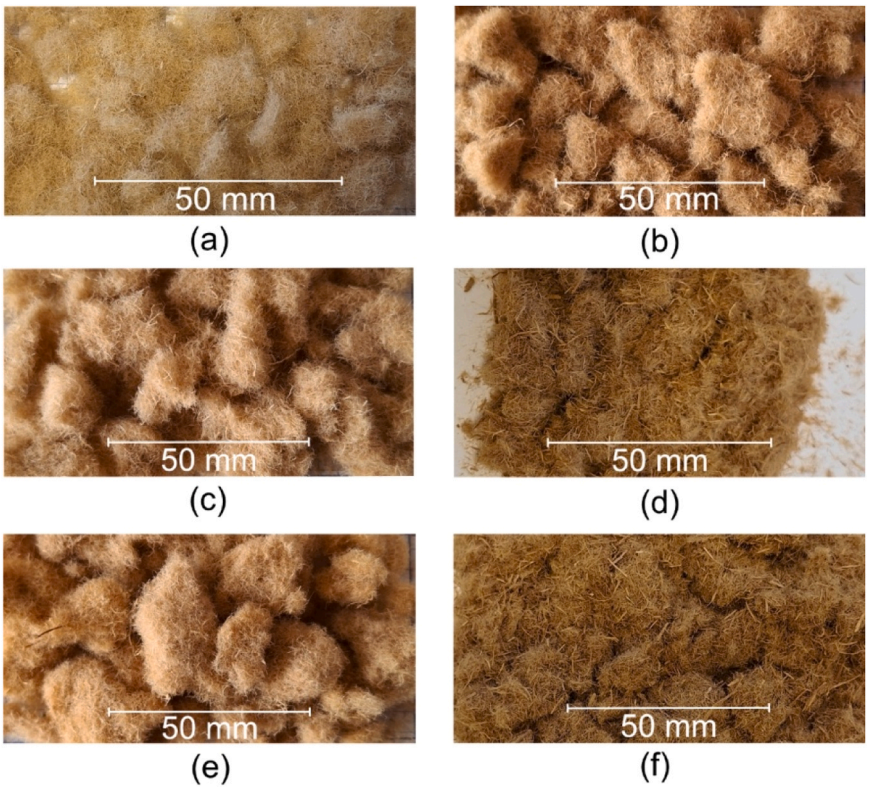

Fig. 2. Virgin and secondary fibre types. (a) virgin fibres, (b) V + 25 %SW, (c) V + 25 %FW, (d) 100 %FW1, (e) V + 25 %PW, (f) 100 %PW1.

Systems (SMS), London, United Kingdom). This approach investigates the sorption behaviour of fibres intended for medium-density fibreboard (MDF) manufacturing. The hygroscopicity of fibres influences the thickness swelling and water absorption of MDF via 24 h cold water immersion (EN 317). The samples were oven-dried before the analysis. Both adsorption and desorption were examined using 20 ± 2 mg of wood dust with relative humidity (RH) from 10 % to 95 % and Temperature of 25 ℃ at steps of 10 % (RH 10–90 %) and 5 % (RH 90–95 %).

2.7. Elemental analysis

The organic elemental analyzer Elementar Vario EL equipped with a TCD detector (Elementar Analysensysteme GmbH, Langenselbold, Germany) measured nitrogen content. The sample (5–50 mg) was sealed and compressed into a tin foil boat before being introduced into the instrument. The sample was combusted at an oven temperature of 950 °C and the resulting gases were reduced on a copper column. Carbon and hydrogen were absorbed on absorption columns while nitrogen was measured.

Elemental analyses were performed using a Thermo Jarrell-Ash IRIS (Duo HR / Advantage) ICP-OES (Thermo Jarrell-Ash, Franklin, MA, USA), by axial or radial viewing of plasma. The samples (~0.3 g of dried) were digested according to "Milestone Application Note Plant Material" with a recommended change of temperature: Digestion of wood samples at 240 °C in concentrated nitric acid assisted using microwave. The digested sample was cooled and diluted with water.

2.8. pH and buffer capacity

The wood fibres were ground to powder < 0.315 mm, and oven-dried at 50 °C for 24 h. Then, 1 g oven-dried materials were immersed in 20 ml of deionized water (pH 7). The mixture was homogenized by agitating with a shaker. Following initial measurement of the pH of the solute, the acid and alkaline buffering capacity were determined by titration with a 0.05 N solution of NaOH and HCl, respectively. Titration was concluded at pH of 10 and 3, respectively. The buffer capacity was then expressed as mEq (H^+) and (OH^-). In this case, the acid and base buffer capacity were calculated based on the equation below:

$$b = \frac{\Delta \quad volume_{\text{acid}} \quad (or\ base)}{\Delta \quad pH_{acid} \quad (or\ base)} \times Normality\ (0.05 \quad N) of\ HCl \quad (or\ NaOH)$$

where Δvolume in ml of HCl/NaOH 0.05 N added up to reach a pH of 3 or 10. Δ*pH* of samples from the initial pH to the target pH of 3 or 10.

To obtain the overall buffering capacity (b_{wa}), the weighted average buffering capacity is estimated as shown below:

$$b_{wa} = \frac{\text{b}_{\text{acid}} \times \Delta pH_{acid} + \quad \text{b}_{\text{base}} \times \Delta pH_{base}}{\Delta pH}$$

where Δ*pH* is the measured ending pH difference from acid to alkaline.

2.9. Residual resin by FTIR

The examination of residual resin on the fibres was done using Fourier transform infrared (FTIR) spectroscopy (UATR Two, PerkinElmer, Shelton, CT, USA). Approximately 0.1 g of finely ground powder was scanned over the wavenumber range of 4000–500 cm^{-1} with a resolution of 4 cm^{-1} , 32 scans per sample.

2.10. Formaldehyde emissions of fibres

The formaldehyde emissions of fibres was measured by the flask method according to EN 717–3 [47].

2.11. Statistical analysis

For statistical analysis, the significance between virgin and

secondary fibres was evaluated using the Kruskal–Wallis test, followed by Dunn's Post-hoc test, with RStudio 2024.04.2 (Posit Software, Boston, United States) at a significance level (α) of 0.05.

3. Results and discussion

3.1. Fibre size distribution

Fig. 3 shows volume-weighted distributions (diameter and length), while Fig. 4 displays mass-based analysis of fibres sieved with an ultrasonic device. Samples with 100 % secondary fibres exhibited a noticeably high ratio of fibres with diameters shorter than 227 μm, and lengths shorter than 1080 μm (Fig. 3a-b, black line). In contrast, fibre blends with 15 % or 25 % substitution rates were comparable to the virgin fibres. Virgin fibre percentile values at × 10, × 50, and × 90 diameters of 99, 402, and 1134 μm respectively. Corresponding values for 100 % secondary fibres from solid wood waste and post-consumer fibreboards were 33 % and 41 % smaller, though the × 90th percentile was 7 % larger. For virgin fibres, percentiles × 10, × 50, and × 90 were 467, 1801, and 3865 μm, respectively, with an average length of 2034 μm. Adding 15 % secondary fibres yielded lengths similar to virgin fibres, except for those from solid wood waste. At 25 % substitution rate, solid wood waste fibres exhibited significantly shorter median and mean fibre lengths, unlike fibres from fibreboard manufacturing residues or post-consumer fibreboards. Notably, the sample V + 25 %PW had a shorter mean fibre length (p-value = 0.01). The median fibre length of 100 % secondary fibres was over 30 % shorter than that of the virgin fibres, regardless of the waste source ($p < 0.0001$). However, 100 % ST fibres retained a mean length comparable to virgin fibres. The difference between median and mean lengths indicates a skewed distribution, suggesting that secondary fibres contain more fines and coarse shives. This is visually apparent in the less uniform distribution in Fig. 3. Fine fibres lower the median, while coarse fibres increase the average length. Previous studies reported that secondary fibres from fibreboard processing residues, treated with steam explosion or hydrothermal processes, were approximately 30 % shorter than virgin fibres used for fibreboard making, due to fibre damage during processing [27,28,48]. Another study by [35] noted a 12 % reduction in mean length when comparing secondary fibres from fibreboard off-cuts to virgin fibreboard fibres. In Fig. 4, the virgin fibres group showed the highest mass fraction of larger fibres (>1240 μm), while fines (<89 μm) were only 25 % compared to 100 % of ST fibres. The high ratio of short fibres observed in Fig. 3 may correspond to this high fine content. Recycled fibres exhibited hemicellulose' degradation and resin residuals that reduce fibre flexibility [30,34,49]. This could make fibres become fragile and further increase the fine content while lowering the larger fractions.

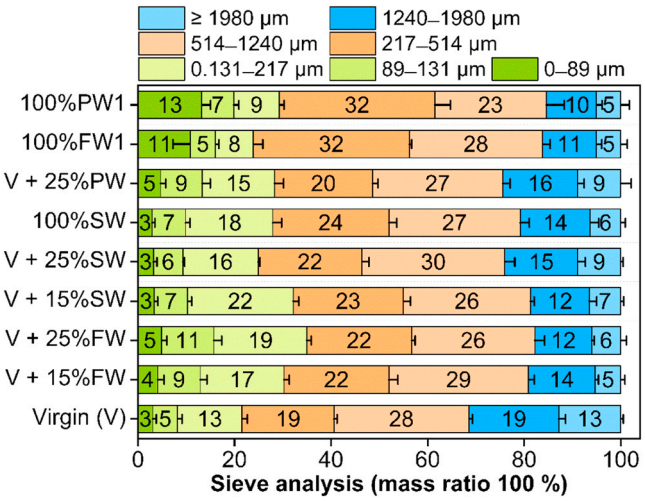

Fig. 4. Sieve analysis of fibres based on mass percentage.

3.2. Fibre morphology

Fig. 5 shows representative SEM images of fibre morphology. All samples exhibited diverse fibre sizes and shapes, ranging from fine, irregular, or spherical broken fibres to unseparated fibre bundles. Virgin fibres had a cleaner surface and less fragmentation compared to secondary fibres. Depending on the fractures, fragments originated from either the primary or secondary cell wall. Sample 100 %SW (Fig. 5b) showed more severe fibre delamination than the other samples. Most fibres retained a rectangular shape without visible fibrillations. However, fibre fibrillations, both external and internal, are known to enhance tensile and internal bond strength by reinforcing the inter-fibre bonding layer [50]. Virgin and secondary fibres displayed characteristic window and border pits typical of pine (*P. sylvestris*). Sample 100 %SW revealed a mix of softwood and hardwood fibres, as indicated by the presence of hardwood vessel elements highlighted in Fig. 5b. Additionally, contaminants were observed in secondary fibres (Fig. 5b–d), appearing as agglomerated balls or irregular sheets. Residual resin and wax were found on secondary fibres from fibreboard waste [35]. These non-fibrous structures likely resulted from fine particles combined with residual resin or other chemical contaminants. The presence of fine

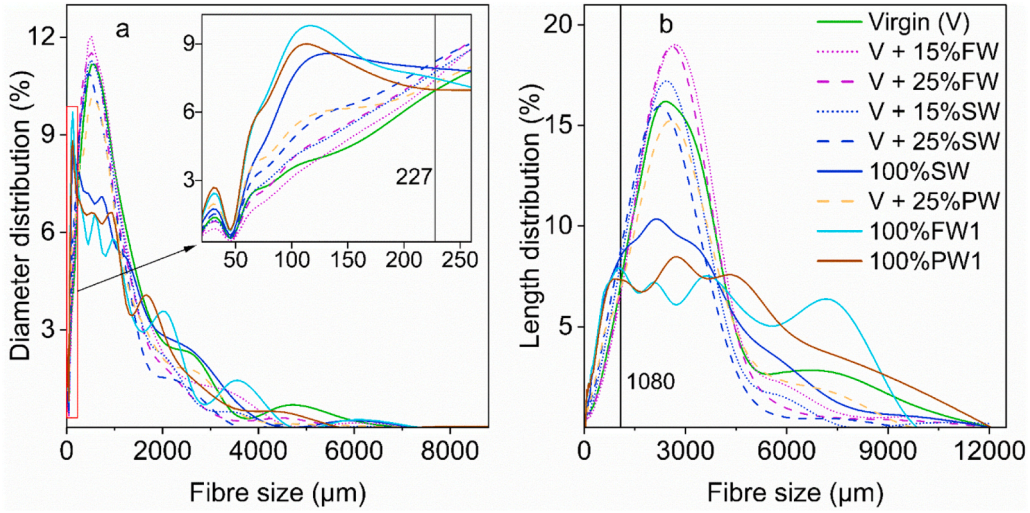

Fig. 3. Particle size distribution by QICPIC. (a) diameter distribution, (b) length distribution.

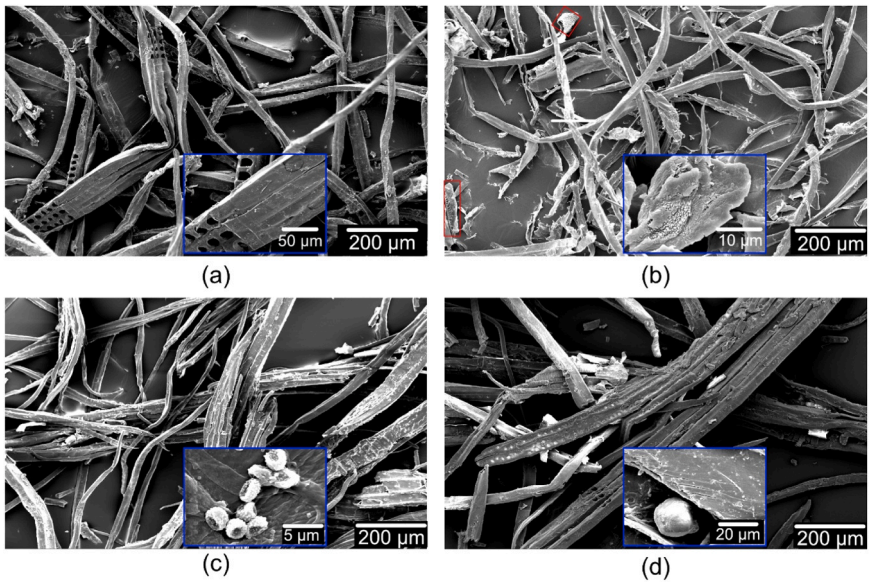

Fig. 5. SEM images of fibres. (a) virgin TMP pine fibres (Virgin), (b) secondary fibres of solid wood waste (100 %SW) with rad blocks showing vessel elements from hardwoods, (c) secondary fibres of fibreboard processing residues (ST-100 %FW1), (d) secondary fibres of post-consumer fibreboards (ST-100 %PW1).

fibres and surface deterioration negatively impacts the bonding capacity of recycled fibres, thereby reducing inter-fibre bond strength [41,51].

3.3. Water sorption

Fig. 6 presents the water sorption, desorption, and hysteresis results at RH ranges from 0 % to 95 %. Fibre samples exhibited equilibrium moisture content (EMC) values ranging from 19.9 % to 21.5 % at 95 % RH, and from 0.3 % to 0.8 % at 0 % RH. Compared to virgin fibres, 100 % secondary fibres from ST are more water-resistant. Virgin materials from Scots pine (*Pinus sylvestris* L.) showed an equilibrium moisture content (EMC) of 20.4 %, which is nearly identical to a previously reported value of 20.5 %. This value is slightly lower than the reported value from Norway spruce (*Picea abies*) [52,53]. The hygroscopic nature of wood fibres and the hysteresis effect are well studied [54–56]. Fibre blends with 15 % or 25 % substitution rates exhibited water sorption behaviour similar to virgin fibres, although the V + 25 %SW showed a slightly lower value. In contrast, sample 100 %SW produced at lower steam pressure (6 bar to 10.4 bar) had water sorption behavior approximately equivalent to the virgin fibres. ST fibres from post-consumer fibreboards and fibreboard processing residues showed significantly lower water sorption (including water retention at 0 % RH) and hysteresis. These changes in secondary fibres were attributed to reduced sorption site accessibility due to degradation of hydroxyl groups, lower hemicellulose content, and the presence of cured residual resin [30,31,57]. Adhesion of wood fibres with UF/MUF resins is primarily enhanced by hydroxyl groups in cellulose and hemicelluloses, which are crucial for hydrogen bonding and condensation via methylol intermediates [58]. The results indicate that 100 % secondary fibres from post-consumer fibreboards are more water-resistant and exhibit a lower bonding affinity to resin than the virgin reference.

3.4. FTIR analysis

The functional groups of fibres were analyzed by FTIR spectra to determine the chemical difference between secondary and virgin fibres (Fig. 7). All fibres exhibited a broad band at 3334 cm^{-1} corresponding to the OH stretching from the hydrogen bond [59]. Virgin pine and mixtures with 15 % and 25 % secondary fibres showed a prominent C–H stretching band at 2899 cm^{-1} . In contrast, secondary fibres from ST and UF-resinated sources exhibited two additional peaks at 2917 cm^{-1} and 2850 cm^{-1} (labels 1,2), corresponding to asymmetric and symmetric stretching of methyl and methylene groups [60,61]. The appearance of the two new peaks is likely due to the presence of wax on the fibres. Additionally, FTIR spectra of resinated virgin fibres showed a primary amide band at 1645 cm^{-1} , attributed to C═O and C–N bonds from UF resin. This peak was shifted to 1657 cm^{-1} for ST fibres, indicating the structural change in the amide group [28,41]. The 1545 cm^{-1} band,

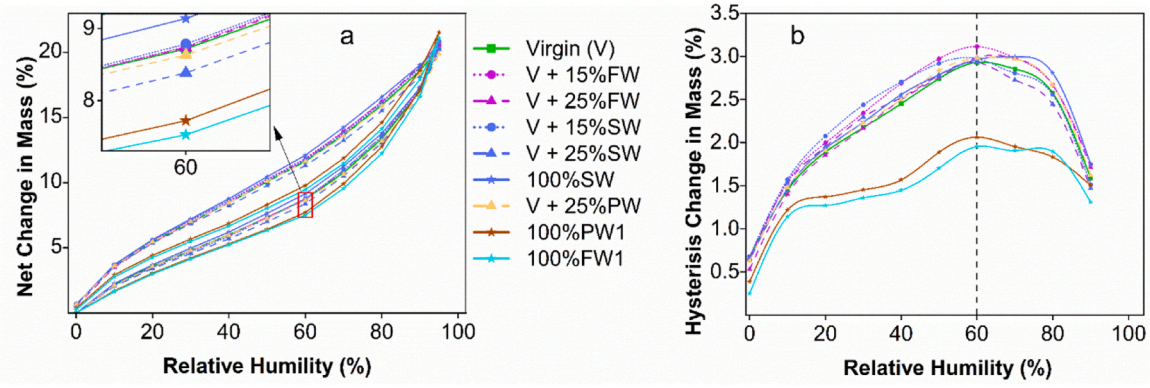

Fig. 6. Dynamic vapour sorption. (a) hysteresis loop, (b) difference between sorption and desorption.

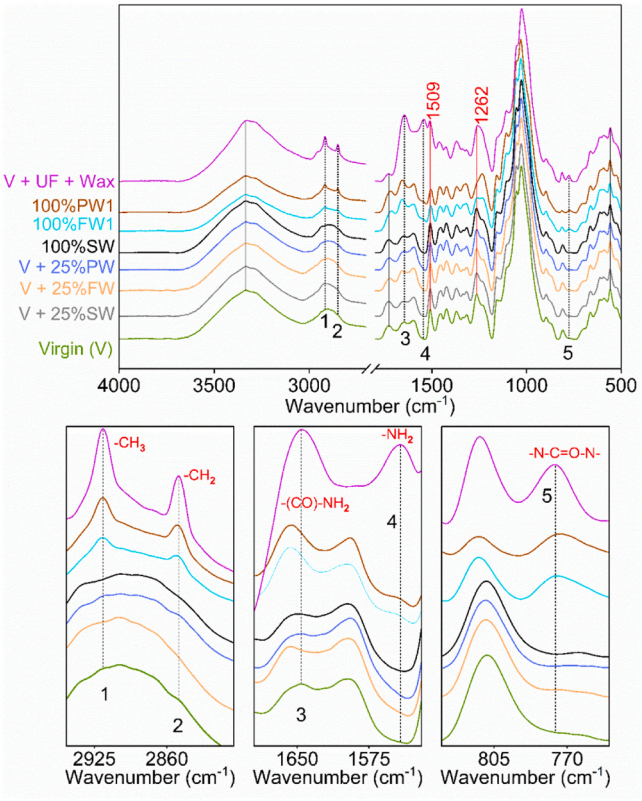

Fig. 7. FTIR results of measured fibre samples.

attributed to C–N stretching of secondary amines, was observed exclusively in UF-resinated fibres [62]. The characteristic peaks at 1730 cm^{-1} and 1509 cm^{-1} were attributed to unconjugated C═O stretching in xylans (hemicelluloses) and aromatic skeletal vibrations, respectively [63,64]. A characteristic absorption band referring to the guaiacyl unit at 1262 cm^{-1} was observed, which is evident in softwoods [65]. The complex peaks and bands ranging from 1457 cm^{-1} to 559 cm^{-1} in the "fingerprint" region are assigned to the main wood components, cellulose, hemicelluloses, and lignin [60,66]. The 100 % ST fibres exhibited a decrease in peak height at 1509 cm^{-1} and 1262 cm^{-1} , indicating the degradation of hemicelluloses and lignin. This finding is consistent with the previously mentioned water sorption results. Additionally, FTIR spectra of cured UF resinated virgin and secondary fibres using ST showed a signal peak at 777 cm^{-1} , typical for C-N deformation of the -N-C═O-N- skeleton [67]. As expected, solid wood fibres showed no chemical evidence of adhesives. However, unexpectedly, fibre blends obtained through the mTMP method also showed no apparent signals. The morphological characteristics of the fibres significantly influence the resultant MDF performance. Panels manufactured with 100 % secondary fibres exhibit a substantial deterioration in physical and mechanical properties. This is primarily attributed to a higher fines content and a reduced fibre slenderness ratio compared to virgin fibres, as well as the presence of residual resin and wax on the recovered fibre surfaces, which may interfere with interfacial bonding. Conversely, MDF incorporating up to 25 % secondary fibres from processing waste maintains acceptable board performance, as demonstrated in our previous research [39].

3.5. Nitrogen content and ICP analysis

Nitrogen content results of the fibre samples are shown in Fig. 8. Virgin Scots pine fibres contained less than 0.2 % nitrogen based on mass, which is consistent with previous studies reporting wood fibre nitrogen levels between 0.1 % and 0.5 % [41,68]. 100 % secondary

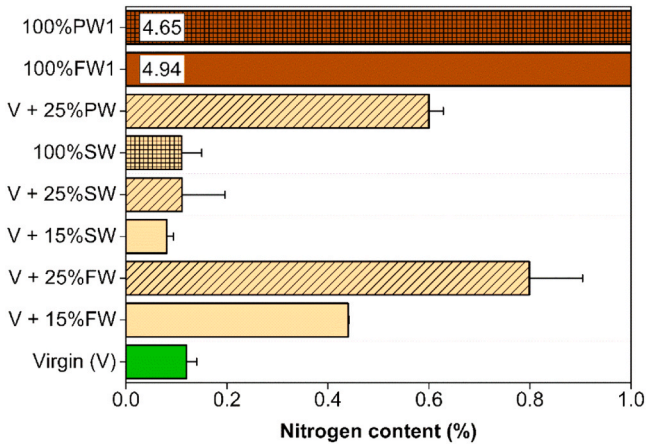

Fig. 8. Nitrogen content of measured fibre samples.

fibres from solid wood waste showed nitrogen levels comparable to virgin fibres. In contrast, fibres from fibreboard processing residues and post-consumer fibreboards exhibited significantly higher nitrogen content, up to 4.9 %, which is nearly 50 times that of the virgin reference. Based on the results of fibre mixtures containing 25 % secondary fibres processed by mTMP, the nitrogen content of 100 % secondary FW and PW fibres reached 2.8 % and 2.0 %, respectively. The measured nitrogen content of a commercial fibreboard can be up to 6.4 % [69]. After steam refining, recycled fibres from fibreboard waste containing 8 % UF resin showed nitrogen contents ranging from 3.6 % to 3.9 %, which is approximately 1 % lower than those obtained by the ST method [28]. The difference presumably comes from the recycling methods, waste source, and the resin load. Higher hydrolysis temperatures and acidic conditions enhance the removal of UF resin from fibreboard waste [29]. The refining process may also help break down the cured resin on the fibre surface through the refiner discs. The high nitrogen content in recycled fibres is attributed to residual resin, which impairs the curing behaviour and bonding strength of the new adhesive system [35,51].

The elemental analysis highlights that low macronutrients (Na, Mg, P, S, K, Ca) were found in low concentrations (< 0.1 μg/g) in the fibre samples. No significant difference was observed between fibres for these elements, except sodium (Table 2), which ranges from 30 to 253 μg/g. ST fibres exhibited significantly higher sodium content than the other samples. Micronutrients (Mn, Fe, Zn, Al, Cu, Co) were present at higher levels (> 1.0 μg/g), although cobalt was near undetectable due to concentrations being lower than the detectable limit (0.2 μg/g). Heavy metals (As, Cd, Ni, Cr, Pb, Ba) were generally absent, except for barium,

Table 2
Trace elements and heavy metals of measured samples by ICP-OES.

Element /Fibre	Na μg/g (11.1)	Mn μg/g (2)	Fe μg/g (4.4)	Al μg/g (4)	Pb μg/g (0.4)	Ba μg/g (0.3)
Virgin (V)	29.4 ± 8.5	67.0 ± 2.8	18.1 ± 11.9	12 ± 1.4	0.8 ± 0.6	2.9 ± 0.1
V + 15 % FW	30.3	77.0	32.9	11.0	0.4	4.1
V + 25 % FW	47.8 ± 4.7	76.7 ± 3.2	69.3 ± 0.1	14.8 ± 0.3	0.4 ± 0.0	4.9 ± 0.4
V + 15 % SW	32.7	73.0	28.4	14.0	0.4	4.2
V + 25 % SW	58.3 ± 4.0	75.8 ± 3.1	41.0 ± 0.4	14.5 ± 0.6	0.4 ± 0.0	5.4 ± 0.4
100 %SW	160.6	75.0	82.8	57.0	2.0	9.5
V + 25 % PW	105.3	64.0	76.3	48.0	1.3	3.3
100 % PW1	253.7 ± 11.8	86.9 ± 3.6	265.7 ± 98.6	203.1 ± 29.7	15.2 ± 1.6	44.7 ± 6.9
100 % FW1	230.9 ± 2.7	97.6 ± 6.3	98.7 ± 20.7	103.1 ± 8.6	12.8 ± 1.5	36.0 ± 7.8

which showed clear signals (> 2.9 μg/g). All the examined fibres contained similar traces of As, Cd, and Ni, which were 0.7 μg/g, 0.2 μg/g, and 1.1 μg/g, respectively. The high heavy metal concentrations in 100 % secondary fibres are likely linked to chemical residues (coatings, pigments, preservatives) and furnishings [4,70]. Compared to previous results, recycled fibres exhibited high content of iron, aluminium, and lead, consistent with the levels in fibreboard waste [69]. As expected, the 100 % secondary fibres from post-consumer fibreboards and fibreboard manufacturing residues exhibited significantly higher chemical composition (particularly sodium, iron, aluminium, lead, and barium), while the levels of solid wood waste fibres were approximately equal to the virgin reference. The chemical levels also increased with a higher substitution rate, compared to the virgin fibres. Moreover, a higher level of sodium cation may react with carbonate anions from the wood adhesive filler, calcium carbonate, added when making panels [71]. This can lead to hydrolysis, releasing hydroxide (OH^-), which contributes to raising the pH. Secondary fibres with a high content of chemical impurities can raise disposal issues, prohibiting their application.

3.6. pH and buffering capacity

Fibres' pH and buffering capacity values are shown in Table 3, with the corresponding titration curve presented in Fig. 9. Virgin fibres had a pH of approximately 4.1, while incorporating secondary fibres reduced acidity, regardless of the waste source. Among fibre blends with 25 % substitution rate, solid wood waste fibres had the lowest pH, similar to virgin fibres, whereas fibres (100 %) recycled via ST showed the least acidic pH. This result of ST fibres aligns with previous studies [30] and is reportedly due to the reduction of fibre acidity from the hydrothermal process. Utilizing steam and pressure, hydrothermal treatment partially degrades hemicelluloses and lignin while removing acidic extractives like phenolic, fatty, and resin acids [72,73]. Adhesives like UF and MUF are alkaline, and their degradation products, such as ammonia, are released during the recycling process, contributing to an increased pH of the recycled fibres [32,33]. ST fibres showed a significant increase in both acid and base buffering capacity. However, no significant difference was observed in fibre mixes with 25 % secondary fibres compared to virgin fibres. Since acid or acid-releasing catalysts are typically used for UF and MUF resins to create an acidic environment for curing, the high pH and buffering capacity of recycled fibres may prolong gel and curing times [30,74]. These results indicate that 100 % secondary fibres by the ST process may require additional acid catalysts and extended hot-pressing time to mitigate the resin curing problems.

3.7. Formaldehyde release

Formaldehyde emission values of fibres are shown in Fig. 10. The virgin Scots pine fibres produced using the TMP process displayed a baseline formaldehyde emission of 5.8 mg/kg, consistent with the reported range of 2–10 mg/kg for Norway spruce and Scots pine under EN 717–3 conditions [75]. This emission level is attributed to the TMP process, which induces thermal degradation of polysaccharides and hydrolysis of lignin [75,76]. Additionally, the high extractive content characteristic of pine wood has been shown to contribute to increased

Table 3
Initial pH and buffer capacity of measured fibres.

Samples	Initial pH	ƀ_acid	ƀ_base	ƀ_capacity
		Unit = mol/L·pH		
Virgin (V)	4.07 ± 0.08	0.045 ± 0.01	0.035 ± 0.01	0.036
V + 25 %FW	4.54 ± 0.04	0.051 ± 0.01	0.040 ± 0.00	0.043
V + 25 %SW	4.12 ± 0.02	0.056 ± 0.01	0.033 ± 0.00	0.037
100 %SW	4.69 ± 0.04	0.054 ± 0.00	0.026 ± 0.00	0.033
V + 25 %PW	4.21 ± 0.03	0.053 ± 0.01	0.047 ± 0.01	0.048
100 %PW1	5.48 ± 0.06	0.056 ± 0.00	0.056 ± 0.01	0.058
100 %FW1	5.42 ± 0.07	0.062 ± 0.00	0.057 ± 0.01	0.060

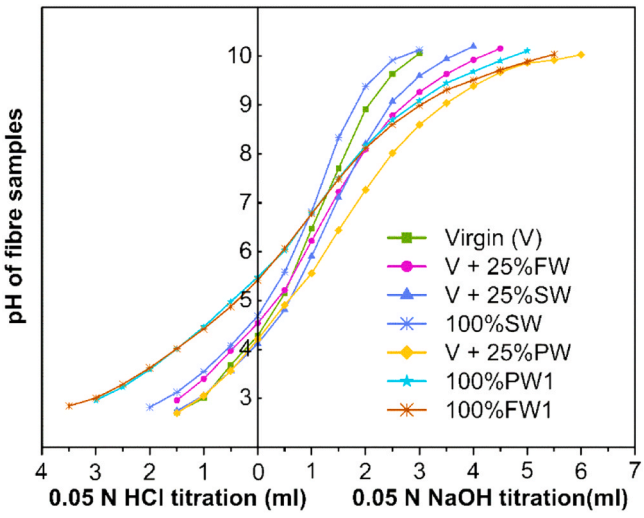

Fig. 9. Acid and alkali titration of fibres.

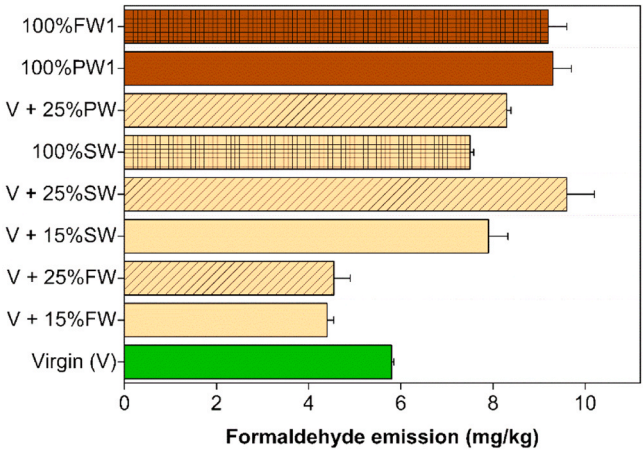

Fig. 10. Formaldehyde emission of fibre samples.

formaldehyde release [75,77]. Sample V + 25 %FW showed a reduction of formaldehyde emissions by 29 %, while emissions increased by 25 % for sample V + 25 % PW, compared to virgin fibres. Secondary fibres (100 %) from the ST process showed a 56 % increase in emissions over virgin fibres. Solid wood waste fibres also exhibited increased emissions, though the results were inconsistent. It was claimed previously that the hydrolysis of methylene-ether linkages of residual resin is a potential source for the subsequent formaldehyde emissions from panels [78]. Overall, incorporating recycled fibres into fibreboards reduced formaldehyde release compared to the virgin TMP fibre reference with UF resin. This decrease is likely due to degradation products, such as urea and ammonia, which react with formaldehyde and act as scavengers [8,42,79]. The increase in formaldehyde emissions might be due to the unhydrolyzed UF resin on recycled fibres [41,80]. However, solid wood waste exhibited higher formaldehyde release at a 25 % substitution rate, while emissions decreased at a 100 % secondary fibre rate. Only 100 % SW fibres were produced using a higher pressure (10 bar vs. 6 bar) by mTMP; these samples underwent greater thermal/hydrolytic degradation, which might have promoted the formation of free formaldehyde or formaldehyde precursors.

4. Conclusions

Secondary fibres (100 % fraction) recovered via modified thermomechanical pulping (mTMP) and steam treatment (ST) from

fibreboard processing residues and post-consumer wood (including fibreboards and solid wood), generally exhibit reduced length, increased width, and higher fines content compared to virgin fibres. However, fibre mixtures with 15 % or 25 % (w/w) secondary fibres show measured properties comparable to virgin fibres. Microscopic characterization revealed that virgin fibres possess superior structural integrity and smoother surface morphologies, whereas secondary fibres show evidence of mechanical degradation. FTIR analysis suggests the presence of residual resin on secondary fibres, which is reflected in the significantly elevated nitrogen content, higher initial pH, and increased pH buffering capacity (acid and alkaline). Notably, while most secondary fibres showed increased alkaline buffering, those derived from solid wood waste displayed reduced alkaline but increased acid buffering capacity. Furthermore, ST fibres from post-consumer fibreboards exhibited lower water sorption and significantly higher micronutrients and heavy metals, as quantified by ICP results. Formaldehyde emissions increase with the proportion of secondary fibre content.

Overall, these findings suggest that up to 25 % of virgin fibres can be substituted with secondary fibres from fibreboard processing residues without significantly compromising fibre quality. Future research will investigate the chemical nature of residual resin, determine the intrinsic tensile strength of secondary fibres, and evaluate the mechanical performance of fibreboard produced with various secondary fibres substitution levels from the mTMP and ST recycling methods.

CRediT authorship contribution statement

Stergios Adamopoulos: Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. **Joran van Blokland:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Triveni Soubam:** Writing – review & editing, Methodology, Data curation. **Percy Alao:** Writing – review & editing, Methodology, Formal analysis, Data curation. **xu Changling:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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