



Antioxidant dietary fibre: A structure-function journey

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ABSTRACT

Background: Non-extractable polyphenols (NEP) are polyphenols that are bound to dietary fibre (DF) and have important health benefits to humans. Past studies have used hydrolysis methods to extract NEP, studying NEP and DF separately. However, hydrolysis may affect their structures and functional properties. In fact, DF is a carrier of NEP in foods, and they physically and/or chemically interact with each other and exist as a complex, known as “antioxidant dietary fibre (ADF)”. Therefore, the review focuses on the natural structure: ADF. The food matrix is a complex system with multiple components, and it is very important to explore approaches to obtain ADF and the effect of its structure on functional properties.

Scope and approach: This review summarizes methods for isolating ADF from samples or forming it *in vitro*, as well as structural characterization, and discusses the functional implications arising from ADF.

Key findings and conclusions: The ADF structure, especially the interactions between NEP and DF, has been characterized. Covalent interactions have been identified in the isolated ADF, and non-covalent interactions are the primary driving force in formed ADF. ADF is bioaccessible and bioavailable, has prebiotic, antioxidant, and anti-inflammatory properties. The structures of ADF, such as the structures and compositions of NEP and DF and their interactions, have a significant role in its functional properties. This review highlights the importance of ADF and provides structure-function information, which will contribute to future studies on its health benefits and the development of functional foods.

1. Introduction

Polyphenols are secondary plant metabolites and have been proven to have health benefits to humans, such as antioxidant, anticancer, antidiabetic, and cardioprotective effects (Aravind, Wichienchot, Tsao, Ramakrishnan, & Chakkaravarthi, 2021). Polyphenols are generally classified into extractable polyphenols and non-extractable polyphenols (NEP) (Wang, Li, Ge, & Lin, 2020). Extractable polyphenols are extracted with aqueous or aqueous-organic solvents. For NEP, mainly high-molecular-weight proanthocyanidins, hydrolysable tannins, phenolic acids, and flavonoids, cannot be extracted because they physically and/or chemically interact with cell wall polymers, mainly dietary fibre (DF) (Pérez-Jiménez & Torres, 2011; Zhang, Zhang, Li, Deng, & Tsao, 2020). In addition, DF is a carrier of NEP during gastrointestinal digestion, they are barely digested by the small intestine and reach the colon, where they are fermented and utilized by colonic microbiota

(Saura-Calixto, 2011). These indicate the fact that NEP and DF are closely related in the food matrix and exist as a complex, known as “antioxidant dietary fibre (ADF)”, which has the characteristics of both NEP and DF (Das et al., 2020; Rocchetti et al., 2022; Saura-Calixto, 2011).

However, previous studies have always investigated NEP and DF separately, using chemical methods (acid and alkaline hydrolysis) or enzymatic methods (e.g., cellulase, xylanase, and β -glucosidase) to treat samples to disrupt their interactions, convert NEP into “extractable polyphenols” (Tang et al., 2016; Wang et al., 2020). The hydrolysis process may alter the original structure of NEP; for instance, alkaline hydrolysis induces the conversion of vanillic acid to vanillin (Fig. 1A) (Pérez-Jiménez & Torres, 2011), resulting in compounds that are mostly derivatives or degradation products of NEP (Martins, Rodrigues, Mercali, & Rodrigues, 2022). Similarly, DF has changes in structure and a significant decrease in its water retention capacity, oil retention

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capacity, and antioxidant activity (Peng et al., 2021; Yin et al., 2022) (Fig. 1B). These results mean that NEP and DF are not independent compounds in the food matrix; they are closely associated with each other and play a specific role when taken as a whole (Pérez-Jiménez & Torres, 2011; Saura-Calixto, 2011). Therefore, we focus on the natural structure: ADF.

The compositions and proportions of NEP and DF vary in the different food matrices; the most abundant NEP and DF in fruits and vegetables are flavonoids and pectins, and in grains, they are phenolic acids and cellulose, which interact with each other through different mechanisms (Das et al., 2023). Phenolic acids tend to form ether and ester bonds with DF through their carboxylic groups and hydroxyl groups (Zhang et al., 2020). Flavonoids mainly interact with DF through hydrogen bonding driven by hydrophilic hydroxyl groups (González-Aguilar, Blancas-Benítez, & Sáysago-Ayerdi, 2017). Proanthocyanidins and tannins can interact with DF by hydrogen bonding or hydrophobic interactions (Hanlin, Hrmova, Harbertson, & Downey, 2010; Pérez-Jiménez & Torres, 2011). In addition, chemical changes may occur in ADF after physical and chemical treatments. For example, Pérez-Jiménez, Díaz-Rubio, Mesías, Morales, and Saura-Calixto (2014) and Silván, Morales, and Saura-Calixto (2010) found that Maillard reaction products interacted with ADF to form new compounds during roasting. The structures of NEP and DF and their interactions, as well as treatments, influence the structures of ADF.

Many studies have investigated the effect of the interactions between NEP and DF on the bioaccessibility and bioavailability of NEP (Palafox-Carlos, Ayala-Zavala, & González-Aguilar, 2011; Rocchetti et al., 2022). However, the structural characterization of ADF and how ADF with different structures affects the exertion of its functional properties has not been systematically discussed. In this review, we aim to summarize methods for obtaining ADF, including isolation and formation methods, investigate its chemical structure and interactions, and discuss the functional implications of ADF with different structures. This review helps establish a better understanding of ADF and provides the basis for developing corresponding functional foods.

2. Isolation and formation of antioxidant dietary fibre

Table 1 summarizes the isolation and formation methods for obtaining ADF. Isolation methods include liquid extraction and enzymatic treatment, while formation methods refer to adsorption and antisolvent precipitation methods.

The liquid extraction method uses aqueous or aqueous-organic solvents to remove extractable polyphenols, proteins, lipids, vitamins and other components as much as possible from the food matrix. However, due to the complexity of the food matrix, liquid extraction does not guarantee enough selectivity, resulting in ADF still containing other substances. On the other hand, the enzymatic method is efficient in isolating ADF with high purity. These two methods can retain the natural structures of NEP and DF and their interactions.

Adsorption and antisolvent precipitation methods use phenolic

compounds and cell wall materials or analogues to form ADF, known as polyphenol-dietary fibre complexes, *in vitro*. These two methods are not only easy to operate but also yield a pure ADF. However, it is worth noting that the formed ADF does not entirely reflect the interaction between NEP and DF in the food matrix but can be employed as a model to infer their binding.

2.1. Isolation methods

2.1.1. Liquid extraction method

The food matrix contains not only NEP and DF but also extractable polyphenols, proteins, lipids, vitamins and other components (Li, Hu, & Zhang, 2021). In previous studies, the extraction of ADF can be achieved by removing interfering substances from samples using aqueous or aqueous-organic solvents (Alves, Lobo, Domingues, Monteiro, & Perone, 2021; Mateos-Martín, Pérez-Jiménez, Fuguet, & Torres, 2012; Maurer et al., 2019). However, in the study of Maurer et al. (2019), the obtained ADF from grape peel was less pure and contained other interfering substances, such as lipids and protein (Table 1). A nearly pure ADF containing approximately 25% NEP was obtained from grape antioxidant dietary fibre in Mateos-Martín et al. (2012). This difference may be related to the properties of the samples, as the grape peel used by Maurer et al. (2019) contains 2.5% NEP and 25.8% DF. Conversely, the grape antioxidant dietary fibre used by Mateos-Martín et al. (2012) contains 14.81% NEP and 73.48% DF. The liquid extraction method is the simplest way to extract ADF but may require further purification.

2.1.2. Enzymatic method

Since DF is the carrier of NEP, ADF can be obtained by extracting DF. The enzymatic method is a common method. α -Amylase, protease, amyloglucosidase, and pancreatin are sequentially employed to treat samples; starch and protein are removed, and extractable polyphenols are also released in this process, yielding soluble and insoluble dietary fibres (Cheng et al., 2017; Yin et al., 2022). Studies have shown that both soluble and insoluble dietary fibres carry NEP, with insoluble dietary fibre tending to carry more NEP (Li, Yang, Li, Huang, & Zhang, 2019; Xu et al., 2020). This means that the “DF” obtained by the enzymatic method is actually the ADF and that interfering substances are removed during the extraction, thus ensuring a higher purity. In addition, a study investigated the ADF in rye bran prepared by the liquid extraction method (80% acetone) and by the enzymatic method. The NEP content in ADF obtained by the enzymatic method was 487.2 mg gallic acid equivalents/100 g, which was significantly higher than that obtained with the liquid extraction method (269.3 mg gallic acid equivalents/100 g) (Iftikhar et al., 2020). This means that the enzymatic method is more efficient in obtaining ADF. However, the isolated ADF may still contain small amounts of extractable polyphenols, starch and protein (Iftikhar et al., 2020; Li et al., 2019) (Table 1); therefore, further purification using solvents was necessary. In conclusion, the enzymatic method improves the purity of ADF and the proportion of NEP, but the experimental procedures are complex and time-consuming, which may

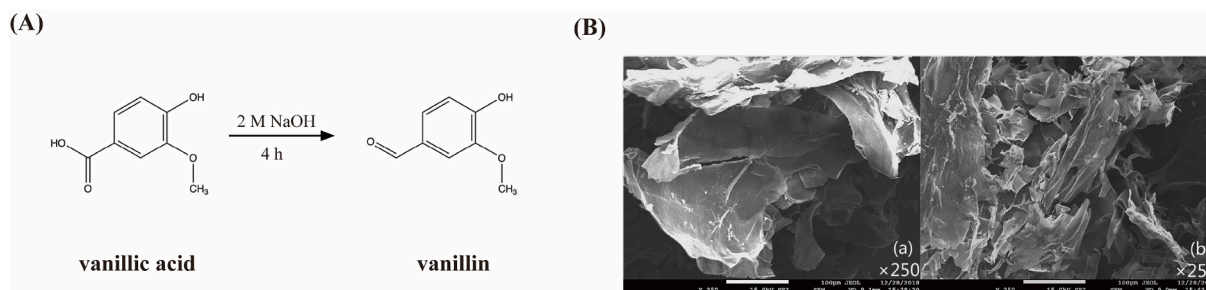


Fig. 1. (A) Conversion of vanillic acid to vanillin during alkaline hydrolysis; (B) scanning electron microscopy images of lychee pulp insoluble dietary fibre before (a) and after (b) removal of non-extractable polyphenols by alkaline hydrolysis (Xu et al., 2020) (Reprinted with permission from the publisher).

Table 1
Isolation and formation methods for antioxidant dietary fibre.

Sources	Isolation and formation methods of ADF	Proximate composition of ADF	Ref.
Liquid extraction method			
Grape peel powder	methanol-water for 60 min and acetone-water for 60 min at room temperature	NEP: 7.7%, DF: 77.6%, lipids: 6.3%, protein: 15.0%	Maurer et al. (2019)
Grape antioxidant dietary fibre	methanol-water and acetone-water-acetic acid at room temperature	NEP: ~25%	Mateos-Martín et al. (2012)
Commercial ground roasted coffee	boiling water for 1 min	NEP: 5278.5 µ g/g	Alves et al. (2021)
Enzymatic method			
Adzuki bean seed coat	α-amylase for 35 min at 95 °C, protease for 60 min at 60 °C, and amyloglucosidase for 60 min at 60 °C	NEP: 61.03 mg gallic acid equivalents/g, DF: 90.51%, starch: 0.1%, protein: 0.23%	Yin et al. (2022)
Lychee pulp	pepsin for 1 h at 40 °C, pancreatin for 4 h at 37 °C, and α-amylase for 16 h at 37 °C		Xu et al. (2020)
Hulless barley flour	α-amylase for 30 min at 95 °C, alcalase protease for 30 min at 45 °C, and amyloglucosidase for 30 min at 65 °C	NEP: 5.90 µ mol ferulic acid equivalents/g soluble dietary fibre, 14.30 µ mol ferulic acid equivalents/g insoluble dietary fibre	Li et al. (2019)
Rye bran	α-amylase for 20 min at 95 °C, alcalase protease for 60 min at 60 °C, and amyloglucosidase for 30 min at 60 °C	NEP: 487.2 mg gallic acid equivalents/100 g, extractable polyphenols: 69.4 mg gallic acid equivalents/100 g, starch: 6.2%, protein: 8.7%	Iftikhar et al. (2020)
Carrot	α-amylase, protease for 30 min at 60 °C, and amyloglucosidase	NEP: 33.03 mg ferulic acid equivalents/10 g, DF: 94.27%, starch: 0.032%, protein: 0.21%	Liu, Jia, et al., 2019a
Rice bran	α-amylase for 20 min at 95 °C, protease for 60 min at 60 °C, and amyloglucosidase for 30 min at 60 °C	NEP: 742.16 mg gallic acid equivalents/100 g	Zhang et al. (2019)
Adsorption method			
Phenolic compounds (hydroxytyrosol, 3,4-dihydroxy-phenylglycol), strawberry cell walls	strawberry cell walls mixed with 1–10 mg/mL polyphenol solutions for overnight		Bermúdez-Oria et al. (2019)
Phenolic compounds (ferulic acid, catechin and cyanidin-3-glucoside), cell wall materials (apple cell walls, cellulose, cellulose-arabinoxylan, cellulose-xyloglucan, and cellulose-pectin)	cell wall materials mixed with 1 mM polyphenol solutions for 1 min–24 h or 2–17 mM polyphenol solutions for 2 h at 4 °C in darkness	maximum content of ferulic acid: 1452 µ g/mg for apple cell walls, 870 µ g/mg for cellulose, 655 µ g/mg for cellulose-arabinoxylan, 1018 µ g/mg for cellulose-xyloglucan, 530 µ g/mg for cellulose-pectin	Phan et al. (2017)
Catechin and cellulose	cellulose mixed with catechin solution (pH 2.0 or 7.0) for 24 h at 37 °C	catechin: 2.70 mg/g (pH 2.0), 2.82 mg/g (pH 7.0)	Liu et al. (2018)
Gallic acid, cell wall materials (oat β-glucans with medium and high molecular weights, guar galactomannan, and xanthan mannoglucuronoglucan)	gallic acid solution mixed with polysaccharide solutions	gallic acid: 175, 230, 27, and 65 gallic acid units every 1000 glycosidic units for oat β-glucans with medium and high molecular weight, guar galactomannan, and xanthan mannoglucuronoglucan	Tudorache et al. (2020)
36 phenolic compounds (flavones, flavonols, flavanones, isoflavones, flavanols, hydroxycinnamic acids, and hydroxybenzoic acids), oat β-glucan	β-glucan mixed with polyphenol solutions for 16 h at 40 °C	phenolic content: flavonol > flvaone > flavanone > isoflavone	Wang et al. (2013)
Phenolic compounds (apigenin, kaempferol, quercetin-3-O-glucoside, pelargonidin-3-O-glucoside, caffeic acid, and strawberry phenolic extract), apple cell walls	apple cell walls mixed with polyphenol solutions for different time (0 or 24 h), pH (2.5 or 6.5), and pressure (processed or unprocessed)		Nagar et al. (2020)
Phenolic compounds (gallic acid and catechin), soluble dietary fibre of lotus root	soluble dietary fibre solution mixed with polyphenol solutions at different pH, temperature and concentration ratio of polysaccharide and polyphenol for 20 min	pH 4, temperature 60 °C and concentration ratio of polysaccharide and polyphenol 4:1, gallic acid: 134.05 mg/g, catechin: 155.74 mg/g	Li et al. (2020)
Tannic acid, bamboo cellulose fibres	bamboo cellulose fibres mixed with tannic acid solution for 24 h at 20 °C at different pH (2, 3.5, 5, 6.5 and 8)	tannic acid: 230 mg/g (pH 5)	Shan et al. (2022)
Phenolic compounds (catechin, gallic acid, ferulic acid, chlorogenic acid and caffeic acid), lotus root polysaccharide	lotus root polysaccharide solution mixed with polyphenol solutions for 30 min at 30 °C	catechin: 61.22 mg/g, gallic acid: 9.37 mg/g, ferulic acid: 29.28 mg/g, chlorogenic acid: 83.80 mg/g, caffeic acid: 14.80 mg/g	Yi et al. (2022)
Antisolvent precipitation method			
Resveratrol, corn soluble dietary fibre	corn soluble dietary fibre was dissolved in water, resveratrol was dissolved in ethanol, then two solutions mixed	resveratrol: 8.29%	Ji et al. (2020)
Quercetin, cellulose	cellulose aqueous solution mixed with quercetin ethanol solution		Khor et al. (2017)
Curcumin, fenugreek soluble dietary fibre	fenugreek soluble dietary fibre was dissolved in water, curcumin ethanol solution with different concentrations was added to the solution		Hu et al. (2023)
Curcumin, soy soluble polysaccharide	soy soluble polysaccharide was dissolved in water and the pH was adjusted to 4.0 or 7.0. Curcumin ethanol solution was added to the solution	curcumin: 4.49 µ g/mg (pH 4.0), 3.41 µ g/mg (pH 7.0)	Chen et al. (2017)

Note: NEP, non-extractable polyphenols; DF, dietary fibre.

lead to a loss of NEP.

2.2. Formation methods

2.2.1. Adsorption method

The plant cell wall is the primary source of DF for human consumption and is mainly composed of cellulose, hemicellulose, and pectin (Siemińska-Kuczer, Szymańska-Chargot, & Zdunek, 2022). Studies have proven that cell wall polysaccharides have an adsorption capacity for polyphenols, and the interaction is mainly the result of hydrophobic effects and hydrogen bonding. Covalent (ester) bonds were also involved in some experiments (Bermúdez-Oria, Rodríguez-Gutiérrez, Fernández-Prior, Vioque, & Fernández-Bolaños, 2019; Zhu, 2018). This method has been utilized to simulate the interactions between polyphenols and polysaccharides in food systems, which helps to understand the mechanism of their interactions and the impact of the interactions on the bioaccessibility of polyphenols in the gut (Phan, Flanagan, D'Arcy, & Gidley, 2017; Zhu, 2018). Thus, the adsorption behaviour of polyphenols onto polysaccharides can be used to form ADF *in vitro*. Apple, grape, and strawberry are common samples for preparing cell wall materials but need to be washed repeatedly with solvents before use to remove endogenous polyphenols, colour, protein, and so on (Bindon, Smith, & Kennedy, 2010; Phan et al., 2017). An adsorption experiment was carried out by soaking cell wall materials in polyphenol solutions or by mixing the two solutions (Liu, Lopez-Sanchez, et al., 2019b). Cell wall components or analogues are also applied for adsorption experiments (Phan et al., 2017; Shan et al., 2022). Studies show that different cell wall materials have different adsorption capacities for phenolic compounds due to the influence of endogenous and exogenous factors (Phan et al., 2015; Tudorache, McDonald, & Bordenave, 2020; Yi et al., 2022). Endogenous factors include the molecular structures, native charge, compositions, and concentrations of cell wall materials and phenolic compounds (Phan et al., 2015, 2017; Wang, Liu, Chen, & Zhao, 2013). Exogenous factors such as processing methods, pH, adsorption time, and temperature also affect the binding of NEP and DF (Liu, Martinez-Sanz, Lopez-Sanchez, Gilbert, & Gidley, 2017; Nagar, Berenshtein, Okun, & Shpigelman, 2020). However, changing those conditions may result in the release of phenolic compounds from cell wall materials (Zhu, 2018). Other researchers have also reported that this adsorption was a relatively reversible process and that phenolic compounds were not immobilized on DF (Phan et al., 2017; Shan et al., 2022). Therefore, this adsorption method, despite being able to form ADF, is limited by numerous factors.

2.2.2. Antisolvent precipitation method

ADF can also be obtained using the antisolvent precipitation method, especially for phenolic compounds with low solubility in water, such as resveratrol, quercetin and curcuminoids (Li, Lin, et al., 2021). The method consists of dissolving polyphenols in organic solvents such as ethanol and isopropanol, whereas DF is dissolved in water. A precipitate is formed when two solutions are mixed because DF is insoluble in organic solvents; under these conditions, polyphenols are encapsulated in DF, resulting in the formation of ADF (Khor, Ng, Chan, & Dong, 2017; Li, Lin, et al., 2021). The method is based on the natural interactions between polyphenols and DF, using DF as a carrier of polyphenols to form ADF (Hu et al., 2023). This method not only improves the water solubility of polyphenols and their absorption and utilization in the gastrointestinal environment but also produces a pure ADF (Li, Hu, & Zhang, 2021; Recharla, Riaz, Ko, & Park, 2017). Previous studies have shown that the formation of ADF is mainly driven by hydrogen bonding, electrostatic forces, or hydrophobic interactions (Chen, Ou, Chen, & Tang, 2017; Hu et al., 2023). Chen et al. (2017) investigated the effect of pH on complexation between soy soluble polysaccharide and curcumin, and the results showed that the maximum loading amount of curcumin (4.49 µg/mg) was found at pH = 4 compared with pH = 7 (3.41 µg/mg). In addition, the ratio of polyphenols to DF and the types of DF affected

the formation of ADF (Hu et al., 2023; Khor et al., 2017).

3. Structures of antioxidant dietary fibre

3.1. Chemical structures

The binding of NEP to DF has attracted attention for decades. However, due to the structural complexity of ADF and the limitations of analytical techniques, it is exceedingly challenging to directly prove the existence of ADF in the food matrix and identify its chemical structure. Table 2 summarizes several studies on the structural identification of ADF. In previous studies, mild acid and alkaline hydrolysis and polysaccharide hydrolase were used to treat samples to isolate and identify acylated NEP oligosaccharides, thereby demonstrating that NEP are linked to DF (Allerdings et al., 2005; Bijalwan, Ali, Kesarwani, Yadav, & Mazumder, 2016; Lequart, Nuzillard, Kurek, & Debeire, 1999). Driselase can also be employed to hydrolyse samples to obtain feruloyl-oligosaccharides. This enzyme is produced by the fungus *Irpex lacteus* and degrades polysaccharides but does not destroy the linkages between ferulic acid and sugar moieties (Smith & Harris, 2001). In addition, Sun, Sun, Wang, Zhu, and Wang (2002) found in wheat, rice, rye, barley straw, and maize stems that *p*-coumaric acid was bound to lignin via ester bonds, whereas the formation of ester and ether bonds was observed in ferulic acid and lignin. Unfortunately, this experiment did not identify the structures of these compounds. The acylated NEP oligosaccharides detected to date have mainly been cinnamoyl-oligosaccharides because hydroxycinnamic acids such as ferulic acid, sinapic acid and *p*-coumaric acid are catalysed by peroxidases or laccases to convert phenolic radicals and form oligomers (dimers, trimers and tetramers), thus participating in the cross-linking between polysaccharides and polysaccharides or between polysaccharides and lignin and forming ester, ether and C–C bonds with DF (Allerdings et al., 2005; Bunzel, 2010).

For ADF formed *in vitro*, as shown in Table 2, the interactions between phenolic compounds and cell wall materials have been confirmed through analytical techniques, but the binding mechanism is still unclear. In previous studies, the authors proposed possible combinations and tentative structures of ADF (Bermúdez-Oria et al., 2019; Hanlin et al., 2010; Wang et al., 2013).

Table 2
Structural identification of antioxidant dietary fibre.

Sources	Experimental methods	Identified interactions between NEP and DF	Ref.
Maize bran insoluble fibre	50 mM trifluoroacetic acid for 3 h at 100 °C	ester bonds	Allerdings et al. (2005)
Millet bran fibre	0.5% NaOH for 3 h at 60 °C		Bijalwan et al. (2016)
Wheat bran	endoxylanase for 24 h at 60 °C	ester bonds	Lequart et al. (1999)
Pineapple cell walls	driselase for 20 h at 37 °C	ester bonds	Smith and Harris (2001)
Lignins from wheat, rice, rye, barley straws, and maize stems	4 M NaOH for 2 h at 170 °C	ester and ether bonds	Sun et al. (2002)
Hydroxytyrosol, strawberry cell walls	adsorption method	hydrogen and ester bonds	Bermúdez-Oria et al. (2019)
Tannin, apple cell walls	adsorption method	hydrogen bonds	Hanlin et al. (2010)
Luteolin, oat β-glucan	adsorption method	hydrogen bonds	Wang et al. (2013)

Note: NEP, non-extractable polyphenols; DF, dietary fibre.

3.2. Structural characterization

Spectroscopy, thermal analysis, molecular simulation, adsorption model, microscopy and other methods are employed to characterize the molecular structure of ADF (mainly the interactions between NEP and DF) and observe its morphological structure.

3.2.1. Spectroscopy

Spectral analysis techniques are based on the interaction between light and a sample and can identify the atomic or molecular structure of the components present in the samples (Rumaling et al., 2022). Common techniques include ultraviolet–visible spectroscopy, Fourier-transform infrared spectroscopy, circular dichroism spectroscopy, fluorescence spectroscopy and nuclear magnetic resonance spectroscopy. In the studies of Liu, Jia, et al. (2019a) and Xu et al. (2020), Fourier-transform infrared spectroscopy was used to characterize the structural features of ADF isolated from samples with dephenolized DF as the control, and the presence of an absorption peak at 1259 cm^{-1} indicated that NEP were bound to DF. For the formed ADF, spectral analysis techniques characterized its structure by comparing the characteristic peaks of polyphenols, DF, and their physical mixture and the corresponding intensities (Chen et al., 2017; Francioso et al., 2017; Hu et al., 2023; Phan et al., 2017). In conclusion, spectroscopy methods can reflect the structural features of ADF and demonstrate the presence of interactions between NEP and DF. However, ultraviolet–visible spectroscopy does not provide precise information about the ADF structure, the binding mechanism of its components and the binding sites (Liu, le Bourvellec, & Renard, 2020), whereas Fourier-transform infrared, circular dichroism, fluorescence and nuclear magnetic resonance spectroscopy can be used to observe conformational changes and explore the binding mechanism (Liu et al., 2020).

3.2.2. Thermal analysis

Thermal analysis methods, including differential scanning calorimetry, isothermal titration calorimetry, and thermogravimetric analysis, can investigate the thermal properties of ADF. Wu et al. (2011) evaluated the thermal properties of the tea polyphenols- β -glucan complex using differential scanning calorimetry, and the endothermic peak of the complex corresponded to $88\text{ }^{\circ}\text{C}$, which were lower than those of β -glucan and physical complex (105 and $110\text{ }^{\circ}\text{C}$). Isothermal titration calorimetry is applied to measure the free energy, enthalpy and entropy in the reaction process. Watrelot, le Bourvellec, Imbert, and Renard (2013) compared and analysed thermodynamic parameters in an adsorption experiment of procyanidins and apple pectin and demonstrated that the formation of the complex was mainly driven by entropy, which may be mainly due to hydrophobic interactions. However, the interaction between procyanidin and arabinans was mainly driven by enthalpy, involving the formation of hydrogen bonds (Fernandes et al., 2020). In the study of Li et al. (2020), thermogravimetric analysis was performed on catechin/gallic acid-soluble dietary fibre complexes. The results showed that the complexes exhibited higher stability (300 and $260\text{ }^{\circ}\text{C}$) than soluble dietary fibre and their corresponding physical mixtures (260 and $230\text{ }^{\circ}\text{C}$), which was related to the formation of hydrogen bonds in the complexes. In summary, the formation of ADF is mainly driven by enthalpy or entropy, and thermal analysis methods allow the measurement of thermodynamic characteristics to study their interactions.

3.2.3. Molecular simulation methods

Molecular simulation methods such as molecular docking and molecular dynamics simulation enable the prediction of the binding modes and affinities of ligands to receptors at the molecular or atomic level (Vidal-Limon, Aguilar-Toalá, & Liceaga, 2022; Yu, Xu, He, & Liang, 2023). Molecular docking reflects a static binding mode, whereas molecular dynamics simulation explores dynamic processes (Yu et al., 2023). In an adsorption test of catechin to cellulose, molecular docking

results showed that catechins preferred to bind to the hydrophilic surface of cellulose, with hydrogen bonds and van der Waals forces as the main binding forces and no formation of chemical bonds (Liu, Ying, Sanguansri, Cai, & Le, 2018). Wang et al. (2022) used a molecular dynamics simulation to dynamically model the binding process of the epicatechin gallate-carboxymethyl β -glucan complex. Their observations revealed that epicatechin gallate bound to the surface of the β -glucan outer helix and may preferentially bind to the carbonyl group located on the 6-glucose residue on its helical surface. Fernandes, Brás, Mateus, and de Freitas (2014) investigated the binding of two anthocyanins, cyanidin-3-O-glucoside and delphinidin-3-O-glucoside, to pectin. The investigation discovered that anthocyanins interacted with pectin through hydrophobic contacts and that delphinidin-3-O-glucoside had a greater binding affinity for pectin than cyanidin-3-O-glucoside due to its B ring possessing more hydroxyl groups. The interactions of charged and neutral delphinidin-3-O-glucoside with pectin were also compared, and the results showed that a positive charge significantly promoted the interaction of delphinidin-3-O-glucoside with sugar rings and hydroxyl and carboxylate groups of pectin and that hydrogen bonds and dispersive contacts were the main binding forces. Therefore, this method can provide further information about the conformation, binding sites and intermolecular force types of ADF on the basis of other analysis techniques (Liu et al., 2018).

3.2.4. Adsorption model

The Langmuir, Freundlich, and Redlich-Peterson isotherm models have been used in adsorption experiments to assess the adsorption capacity of cell wall materials for polyphenols and to analyse their interactions (Liu, Lopez-Sanchez, et al., 2019b; Phan et al., 2015). In the study of Shan et al. (2022), the results of the Langmuir and Freundlich models showed that the adsorption of tannic acid into bamboo cellulose was mainly driven by hydrogen bonding and that the process was reversible. Wang et al. (2013) found that the chemical structures of polyphenols affected their interaction with oat β -glucan and that methylation and methoxylation of phenolic acids reduced their ability to bind to β -glucan. In addition, Renard, Baron, Guyot, and Drilleau (2001) carried out a desorption experiment on procyanidins by washing the procyanidins-apple cell wall complex and found that there were weak interactions between them.

3.2.5. Microscopy

Microscopy enables the visualization of microstructures such as tissues, cells and molecules, and scanning electron microscopy, confocal laser scanning microscopy, atomic force microscopy, and transmission electron microscopy capture 3D volume images used for structural characterization of samples (Liu et al., 2021). Fig. 2 presents scanning electron microscopy images of ADF obtained by enzymatic, adsorption and antisolvent precipitation methods. As shown in Fig. 2A, the dephenolized DF showed a looser overall structure than ADF isolated from carrots (Liu, Jia, et al., 2019a). During the formation of the tannic acid-bamboo cellulose complex, tannic acid agglomerated on the surface of cellulose and formed large particles, which blocked some holes in cellulose (Fig. 2B) (Shan et al., 2022). Ji, Jia, Cao, Muhoza, and Zhang (2020) also observed that in the complex of resveratrol-corn soluble dietary fibre, many resveratrol particles were embedded in dietary fibre (Fig. 2C). Polyphenols possess fluorescence properties, and confocal laser scanning microscopy can be used to visualize ADF structure. Fig. 3A shows that fluorescences of both NEP (green colour) and DF (red colour) were observed in ADF isolated from adzuki bean seed coat (Yin et al., 2022). In an adsorption experiment of ferulic acid with apple cell wall, it was observed that the complex showed green fluorescence (Fig. 3B), suggesting that polyphenols were bound to apple cell wall (Phan et al., 2017). Yi et al. (2022) evaluated the size and morphological features of complexes of phenolics and lotus root polysaccharides using atomic force microscopy. The results indicated that phenolic binding not

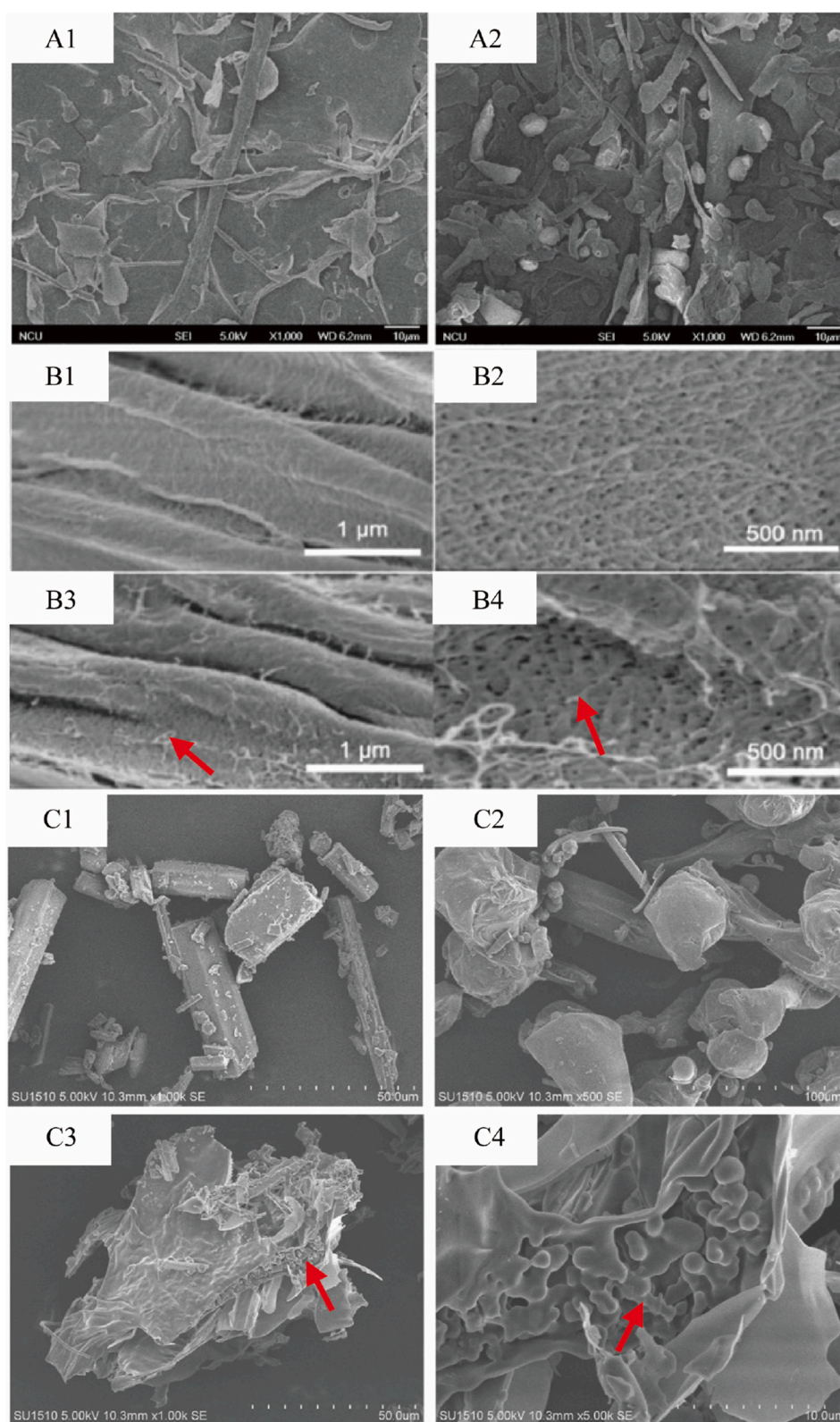


Fig. 2. Scanning electron microscopy images of antioxidant dietary fibre. (A) Enzymatic method, dephenolized dietary fibre (A1) and isolated antioxidant dietary fibre from carrots (A2) (Liu, Jia, et al., 2019a); (B) adsorption method, bamboo cellulose fibre (B1–B2) and tannin acid-bamboo cellulose fibre complex (B3–B4) (Shan et al., 2022); (C) antisolvent precipitation method, resveratrol (C1), corn soluble dietary fibre (C2), and resveratrol-corn soluble dietary fibre complex (C3–C4), voltage 5 kV (Ji et al., 2020) (Reprinted with permission from the publisher).

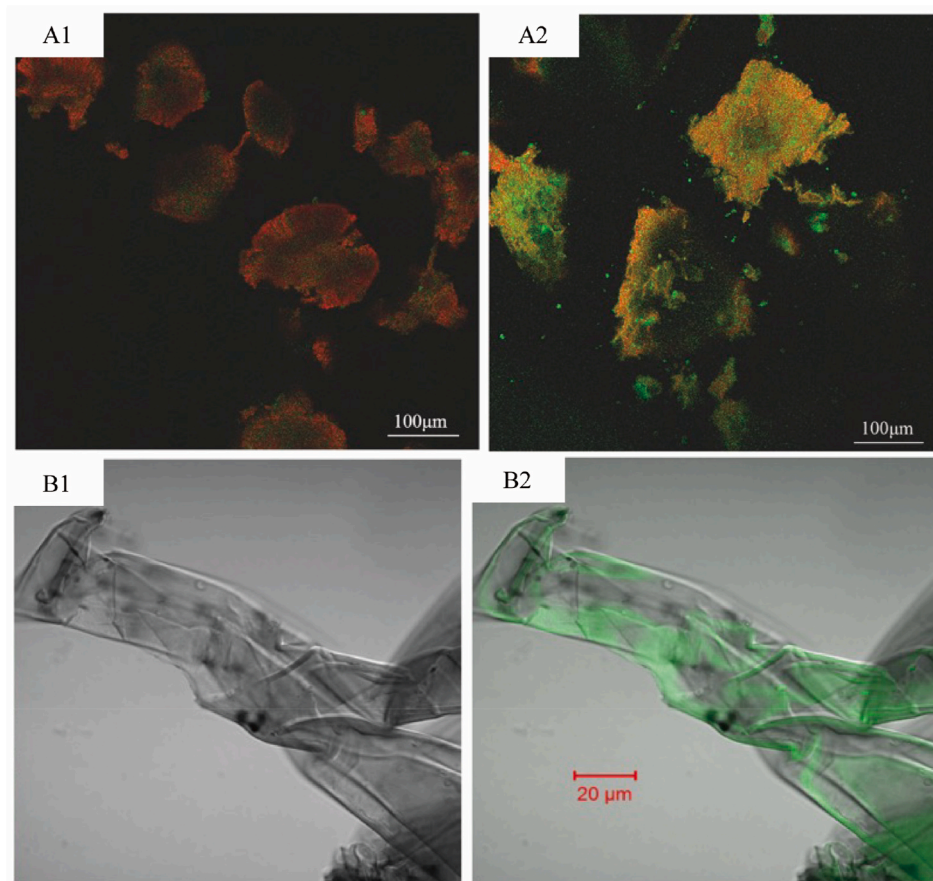


Fig. 3. Confocal laser scanning microscopy images of antioxidant dietary fibre. (A) Enzymatic method, dephenolized dietary fibre (A1) and isolated antioxidant dietary fibre from adzuki bean seed coat (A2), excitation wavelengths: 488 and 408 nm (Yin et al., 2022); (B) adsorption method, apple cell wall (B1) and ferulic acid-apple cell wall complex (B2), excitation wavelength: 405 nm (Phan et al., 2017) (Reprinted with permission from the publisher).

only led to an increase in the average molecular weight of polysaccharides but also significantly affected their molecular conformation. In the study of Hu et al. (2023), transmission electron microscopy observed that curcumin bound to fenugreek soluble dietary fibre precipitated by 60% ethanol to form the complex. Therefore, microscopy methods are capable of directly observing the formation of ADF and its morphological structure, but they need to be combined with other methods to study the interaction of NEP and DF (Liu et al., 2020).

3.2.6. Other methods

X-ray diffraction (Ji et al., 2020), small angle X-ray scattering (Liu, Lopez-Sanchez, et al., 2019b), small angle neutron scattering (Liu et al., 2017), molecular weight changes (Yi et al., 2022) and dynamic light scattering (Chen et al., 2017) have also been used to identify the structure of ADF. Each method only provides fragmentary information, and it is difficult to confirm and characterize the structure of ADF by a single method, so multiple methods have been used in previous studies (Li et al., 2020; Watrelot et al., 2013).

In summary, for the isolated ADF, the above methods did not directly demonstrate the types of interaction between NEP and DF. As we predicted, because NEP contains many phenolic compounds and DF contains cellulose, hemicellulose, etc., the ADF structure is very complex. Current studies characterize the interactions by identifying the structure of acylated NEP oligosaccharides. The interactions identified to date are mainly covalent bonds, and it is speculated that the weak interactions between them may be disrupted during the extraction of oligosaccharides, which needs further exploration. Polyphenols and DF in formed ADF are mainly linked through non-covalent interactions. The complexes formed *in vitro* are of great interest for future studies on how

polyphenols are released from DF and the effect of different interactions on their release.

4. Functional implications of antioxidant dietary fibre

4.1. Bioaccessibility

NEP are gradually released from ADF during oral digestion, gastric digestion, small intestine digestion and colonic fermentation after consumption (Wang et al., 2020). The *in vitro* gastrointestinal model is used to assess the bioaccessibility of NEP in the gut (Dong et al., 2020). In the study of Zhang et al. (2023), the polyphenol content released from the ADF of wheat bran during oral, gastric, and small intestine digestion was 0.30, 0.84 and 0.76 mg gallic acid equivalents/10 g, respectively, and increased to 5.36 mg gallic acid equivalents/10 g at 6 h fermentation. Confocal laser scanning microscopy images were used to visualize the release processes of NEP in rice bran; it was observed that the NEP signal (red colour) was significantly decreased after colonic fermentation (Fig. 4) (Zhang et al., 2019). For the complexes formed by the adsorption method, the release of polyphenols in the gastrointestinal tract was influenced by endogenous and exogenous factors (such as the structures of polyphenols and DF and experimental conditions) (Bermúdez-Oria et al., 2019; García-Pérez et al., 2024; Nagar et al., 2020). For example, the bioaccessibilities of phenolic acids in cherry laurel polyphenols-low-methoxy pectin complex were 6.3% and 80.9% in the small intestine and colon, respectively. In contrast, the bioaccessibilities in cherry laurel polyphenols-high-methoxy pectin complex were 12.6% and 60.7%, respectively (García-Pérez et al., 2024). The curcumin-soy soluble polysaccharide complex obtained by the antisolvent

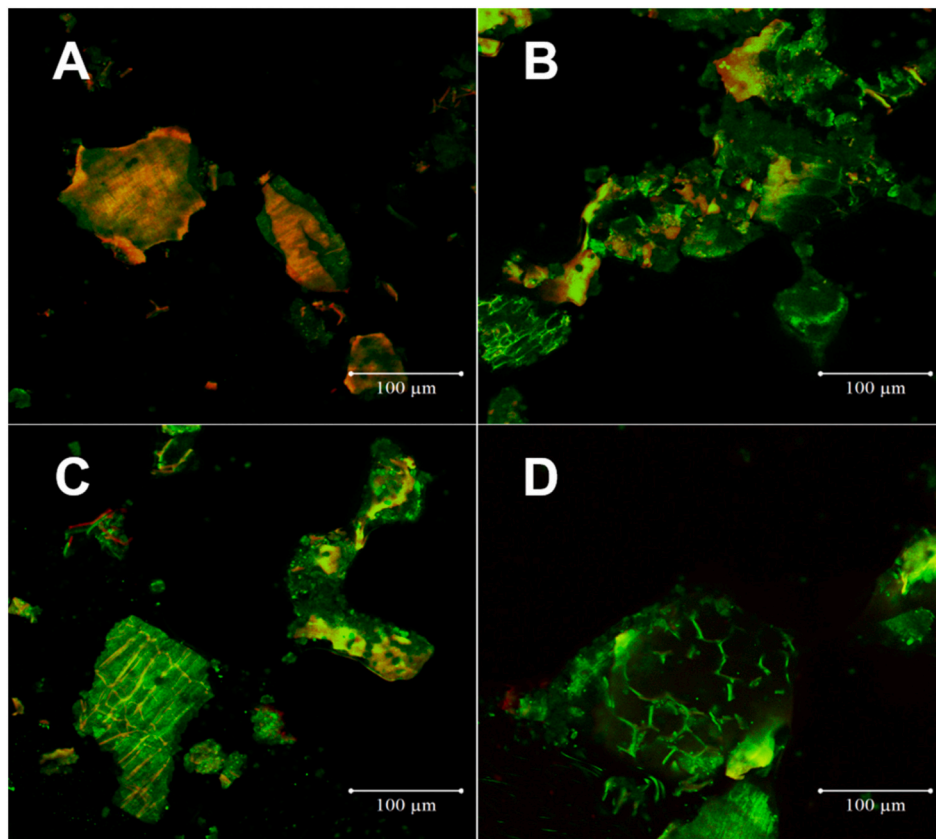


Fig. 4. Confocal laser scanning microscopy images of antioxidant dietary fibre in rice bran without digestion (A), with gastric digestion (B), gastrointestinal digestion (C), gastrointestinal digestion and colon fermentation (D), excitation wavelength: 488 nm, non-extractable polyphenols (red colour) and dietary fibre (green colour) (Zhang et al., 2019) (Reprinted with permission from the publisher). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

precipitation method was quickly released and had high bioaccessibility in the stomach and small intestine (Chen et al., 2017; Ji et al., 2020). Notably, the formed ADF does not represent the actual substrates consumed from foods. In summary, the ADF obtained by different methods may have different bioaccessibilities in the gastrointestinal tract. Unfortunately, there are no studies on the impact of methods and the types of interaction on the release of polyphenols.

4.2. Colonic transformations and bioavailability

In the colon, colon microbiota (such as *Bifidobacterium* spp. and *Lactobacillus* spp.) and enzymes they secrete destroy the NEP and DF interactions and release phenolic compounds and DF, which are further transformed and metabolized by colon microbiota (Pérez-Jiménez, Díaz-Rubio, & Saura-Calixto, 2013; Zhang et al., 2020). The transformation of phenolic compounds involves phase I and/or phase II reactions. In the phase I stage, phenolic compounds undergo deglycosylation in the presence of glycosidases. In phase II, phenolic compounds undergo esterification, decarboxylation, amination, halogenation and hydroxylation reactions, resulting in the formation of simpler phenolic compounds or phenolic metabolites (Gulsunoglu-Konuskan & Kilic-Akyilmaz, 2022; Rocchetti et al., 2022). Compared with extractable polyphenols, NEP need more time to be fermented and transformed, thus exhibiting a delayed absorption time (Das et al., 2023; Pérez-Jiménez et al., 2013). Rondini et al. (2004) also found that the ferulic acid bound to arabinoxylans in bran has a higher residence time in the body compared to free ferulic acid. DF undergoes pathways such as the Wood-Ljungdahl, succinate, and lysine pathways in colonic fermentation, forming short-chain fatty acids (Koh, de Vadder, Kovatcheva-Datchary, & Bäckhed, 2016). Loo et al. (2023) found that *in*

vitro colonic fermentation, the complexes of diosmin-sugarcane fibre, luteolin-sugarcane fibre, and triclin-sugarcane fibre significantly increased the production of short-chain fatty acids than individual flavones and produced beneficial phenolic metabolites such as 3-(3-hydroxyphenyl)-propanoic acid and 3-(3,4-dihydroxyphenyl)-propionic acid compared to fibre. ADF is transformed in the colon to produce phenolic metabolites and short-chain fatty acids through their respective metabolic pathways.

The metabolites produced in the colon are absorbed by enterocytes and hepatocytes, enter the blood circulation exert systemic effects, and are ultimately excreted through urine (Aravind et al., 2021; Das et al., 2023). For example, Lahtinen et al. (2023) found phenolic metabolites (such as ferulic and sinapic acids and their sulfate and glucuronide derivatives) in the urine of rats fed ADF from birch. Nishijima, Takida, Saito, Ikeda, and Iwai (2015) investigated the bioavailabilities of quercetin, pectins (high-methoxy pectin and low-methoxy pectin) and their complexes in humans, and the results showed that quercetin in the complexes exhibited higher bioavailability than free quercetin, with a significant increase (2.5-fold) in metabolites detected in urine, and found that the methylation degree of pectin affected the absorption of quercetin in humans.

4.3. Prebiotic properties

ADF reaches the colon and is extensively fermented by colon microbiota (Saura-Calixto, 2011). Previous studies showed that the intake of ADF alters the types and amounts of microbiota, stimulating the growth of beneficial microbiota (such as *Bacteroidetes*, *Lactobacillus* spp., *Akkermansia* and *Faecalibacterium*) and reducing the *Firmicutes* to *Bacteroidetes* (F/B) ratio, thus showing prebiotic properties (Dong et al.,

2020; Zhang et al., 2019, 2023). Su, Fu, Huang, Liu, and Li (2022) found that the ADF of *Rosa roxburghii* fruit pomace exhibited stronger prebiotic properties than dephenolized DF. Zhang et al. (2019) also reported similar results in rice bran. Table 3 presents changes in colon microbiota after fermentation of the isolated ADF and corresponding interactions. Studies have shown that the intake of ADF stimulates the growth of beneficial microbiota that are associated with the production of glycosidases and ferulic acid esterase, and these enzymes are involved in the degradation of ADF during colonic fermentation (Hughes et al., 2007; Lin et al., 2023; Snelders et al., 2014; Vardakou et al., 2008). The study of Vardakou, Palop, Gasson, Narbad, and Christakopoulos (2007) showed that the presence of ester bonds influenced the production of ferulic acid esterase. In addition, it was observed that arabinoxylans with different molecular weights selectively stimulated microbiota growth. Furthermore, Grant et al. (2020) investigated the effects of the ferulic acid-apple cell wall, catechin-apple cell wall, and cyanidin-3-glucoside-apple cell wall complexes on porcine faecal bacterial populations and found that three complexes promoted the growth of bacteria that were able to utilize the substrates provided, thus causing differences in bacterial population structure. García-Pérez et al. (2024) also found that different types of pectin in the complex may selectively mediate the growth of different microorganism communities. In conclusion, NEP and DF may have a synergistic effect in regulating colon microbiota, and their structures (such as the types of NEP and DF and their interactions) also have significant impacts on microbiota.

4.4. Antioxidant properties

The isolated and formed ADF possesses antioxidant capacity (Khor et al., 2017; Liu, Jia, et al., 2019a; Yi et al., 2022). After the intake of ADF, phenolic compounds are gradually released in the gut. Studies have shown that phenolic compounds released from colonic fermentation show stronger antioxidant activity than in oral, gastric and small intestine digestion, and changes in plasma antioxidant capacity have also been observed (Dong et al., 2020; Pérez-Jiménez et al., 2009; Rondini et al., 2004; Zhang et al., 2023). López-Oliva, Agis-Torres, Goñi, and Muñoz-Martínez (2010) found that ADF was involved in the regulation of the glutathione redox system by increasing the ratio of glutathione to oxidized glutathione and enhancing the scavenging capacity of endogenous antioxidant enzymes, including glutathione peroxidase and peroxisomal catalase, against hydrogen peroxide, which helped maintain intestinal redox homeostasis; thus, antioxidant action in the colonic mucosa of rats was displayed. In the study of Wu et al. (2011), the tea polyphenols-β-glucan complex had higher superoxide dismutase and glutathione peroxidase activities in the liver than the physical mixture of tea polyphenols and β-glucan, which may be related to the formation of hydrogen bonds in the complex. Therefore, the intake of ADF might mitigate intestinal diseases such as inflammatory bowel disease and colorectal cancer caused by oxidative stress by reducing the harmful effects of free radicals in humans (Mosele, Macià, & Motilva, 2015).

Table 3
Changes in colon microbiota after colonic fermentation of antioxidant dietary fibre.

Antioxidant dietary fibre	NEP and DF interactions	Colon microbiota changes	Ref.
Insoluble feruloylated arabinoxylan from wheat flour	ester bonds	<i>Bifidobacteriaceae</i> ↑, <i>Lactobacilli</i> ↑	Vardakou et al. (2008)
Medium and high feruloylated glucuronoarabinoxylans from corn bran	ester bonds	<i>Bifidobacteriaceae</i> ↑, <i>Prevotellaceae</i> ↑, <i>Acidaminococcaceae</i> ↑, <i>Oscillospiraceae</i> ↓	Lin et al. (2023)
Feruloylated arabinoxylan with different molecular masses (354, 278, and 66 kDa) from wheat	ester bonds	<i>Bifidobacteriaceae</i> ↑ (arabinoxylan with 354, 278, and 66 kDa), <i>Lactobacilli</i> ↑ (arabinoxylan with 66 kDa)	Hughes et al. (2007)
Feruloylated arabinoxylanoligosaccharides	ester bonds	<i>Bifidobacteriaceae</i> ↑	Snelders et al. (2014)

Note: NEP, non-extractable polyphenols; DF, dietary fibre.

4.5. Anti-inflammatory properties

Inflammatory bowel disease, including ulcerative colitis and Crohn's disease, is a multifactorial intestinal disease that is related to genetics, the environment, and immunity and manifests clinically as abdominal pain and diarrhoea (Tan et al., 2023). Ulcerative colitis, in particular, specifically occurs in the colon (Tang, Fang, & Ng, 2020). Inflammatory bowel disease may develop into a more serious disease, colorectal cancer (Mosele et al., 2015). Since one of the action sites of ADF is in the colon (Pérez-Jiménez et al., 2013), it is considered to have a potential effect on preventing or treating inflammatory bowel disease. For example, in the studies of Maurer et al. (2019), Maurer, Cazarin, Quatrin, Minuzzi, et al. (2020a) and Maurer, Cazarin, Quatrin, Nichelle, et al. (2020b), the ADF isolated from grapes may alleviate 2,4,6-trinitrobenzene sulfonic acid-induced colon damage compared to DF through different mechanisms, such as improving intestinal barrier function, maintaining intestinal redox homeostasis, and restoring antioxidant enzyme activities.

Overall, NEP and DF exert synergistic/complementary effects and yet other independent effects *in vivo*, and their colonic fermentation and the production of metabolites have important health implications for humans. The ADF structure, such as the structures of NEP and DF and their interactions, has a significant role in its bioaccessibility, colonic transformations and bioavailability in the body.

5. Conclusion and perspectives

Growing evidence indicates that ADF has potential health benefits. Extraction of ADF from the food matrix faces many problems, especially how to reduce the interference of other components and improve purity. These problems can be avoided in the formed ADF. However, the structural identification and characterization of ADF still face challenges. Future studies should focus on the structure of ADF, including the covalent and non-covalent binding mechanism of NEP and DF, and the effect of the structures of NEP and DF on their interactions.

ADF is bioaccessible and bioavailable in the body and has important functional implications in improving human health. The isolated and formed ADF appear to have different bioaccessibilities, and it is advisable to explore further and compare the effects of isolation and formation methods of ADF and interactions between NEP and DF on the digestion, colonic transformations and functional properties of ADF. This review helps us to understand ADF and provides directions for future research.

Author contributions

Xueqing Wang: Conceptualization, Methodology, Writing - original draft; Giorgia Purcaro: Supervision, Writing - review & editing; Bei Fan: Formal analysis, Resources; Li-Tao Tong: Resources, Validation; Liya Liu: Resources, Validation; Jing Sun: Resources, Validation; Fengzhong Wang: Funding acquisition, Project administration, Supervision; Lili

Wang: Conceptualization, Supervision, Writing - review & editing.

Declaration of competing interest

The authors state that they have no conflict of interest.

Data availability

Data will be made available on request.

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