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Review

Analytical approaches to bioactive compounds in Korean agri-food byproducts: A review

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Abstract: Agri-food byproducts in Korea are increasingly investigated as secondary sources of bioactive compounds. However, variability in pretreatment, extraction, and analytical workflows limits comparability across studies. Reliable identification and quantification therefore require robust analytical strategies and standardized validation practices. This review summarizes analytical approaches applied to representative Korean agri-food byproducts, with emphasis on sample pretreatment, extraction, chromatographic analysis, spectroscopic screening, and method validation. Liquid chromatography remains the principal platform for phenolic acids, flavonoids, isoflavones, lignans, and ginsenoside-related compounds, whereas gas chromatography is primarily used for volatile and aroma-active constituents. Spectrophotometric assays and near- or mid-infrared methods provide rapid screening options but show lower selectivity than chromatographic techniques. Across the reviewed studies, analytical results are strongly influenced by matrix-specific pretreatment, including drying, defatting, hydrolysis, and solvent selection. Improved comparability would benefit from matrix-aware validation and more consistent reporting of pretreatment conditions and analytical performance.

Keywords: agri-food byproduct, bioactive compounds, extraction, food analysis

1. Introduction

Growing concern over resource efficiency and waste reduction has increased interest in the sustainable utilization of agri-food byproducts. Within a circular bioeconomy framework, recovery of bioactive compounds from processing residues is considered a practical strategy for value addition and waste minimization.[1,2] As a result, agri-food byproducts are increasingly regarded as secondary sources of bioactive compounds rather than simple waste. Peels, pomace, bran, seeds, and other processing residues may retain phenolic compounds and related phytochemicals relevant to food, nutraceutical, and cosmetic applications.[1-3]

In Korea, diverse agri-food byproducts are generated from the processing of cereals, legumes, fruits, vegetables, oilseeds, and medicinal plants. Representative matrices include rice bran, soybean processing residues, citrus peels and pomace, onion skins, sesame or perilla

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residues, and ginseng marc. The chemical composition of these materials varies with raw material type, processing history, moisture level, lipid content, and storage conditions. In addition, some target compounds occur in bound or conjugated forms, whereas others are volatile or unstable during handling. Therefore, reliable analytical characterization is required before further utilization can be achieved.[2,3]

The analytical value of a byproduct study depends not only on compound yield but also on the quality of sample pretreatment, extraction, separation, and identification. Previous reviews have shown that extraction conditions strongly influence recovery, selectivity, and degradation of phenolic compounds in complex food and plant matrices.[2,3] For non-volatile compounds, UHPLC- and LC–MS-based methods have become central tools because they provide improved selectivity, sensitivity, and structural information for complex samples.[4-6] By contrast, analysis of volatile compounds typically relies on isolation, separation, and identification workflows based on gas chromatography.[7-10] Spectrophotometric assays remain useful for rapid comparison of extracts but provide only global indices with limited specificity.[11-13]

Rapid screening tools have also attracted attention. Infrared and near-infrared spectroscopy enable fast, non-destructive measurement with minimal sample preparation. However, model performance depends strongly on matrix characteristics, analyte concentration, calibration design, and external validation.[14-16] In parallel, several Korean studies continue to apply targeted marker analysis to citrus flavonoids, ginsenosides, and volatile compounds in plant and food matrices.[8,17-19]

Against this background, this review summarizes analytical approaches applied to bioactive compounds in Korean agri-food byproducts, with emphasis on representative matrices, sample pretreatment, extraction strategies, LC-based analysis of non-volatile compounds, GC-based analysis of volatile compounds, rapid screening methods, and practical aspects of method validation. Rather than surveying analytical methods in general, this review highlights how these workflows have been applied to representative Korean byproduct matrices and summarizes reporting and validation items that can improve comparability across studies.

2. Representative Korean Agri-food Byproducts and Target Compounds

2.1. Representative byproducts

Previous Korean studies have reported several representative agri-food byproducts as analytical targets (*Table* 1). These include rice bran, soybean processing

Table 1. Representative Korean agri-food byproducts, principal target compounds, and analytical notes

Byproduct	Principal target compounds	Analytical note	Ref.
Rice bran	Phenolic acids, γ-oryzanol, vitamin E	Bound and free phenolic fractions should be distinguished. Hydrolysis and cleanup can change the observed profile.	[20,21]
Soybean processing residues	Isoflavones	Reported values depend strongly on hydrolysis conditions and sample-to-acid ratio.	[22]
Citrus peel/pomace	Hesperidin, narirutin, methoxylated and glycosylated flavonoids, volatiles	Peel-rich matrices are more relevant than pulp-rich residues for flavonoid-oriented studies.	[17,18,23-25]
Onion skin	Quercetin and related flavonoids	The non-edible outer fractions contain the highest marker levels.	[26,33]
Sesame/perilla residues	Sesamin, sesamolin, phytosterols, broad phenolic fractions	Oilseed residues may require attention to both lipophilic and polar fractions.	[27,28]
Ginseng marc	Ginsenosides, acidic polysaccharides, less-polar metabolites	Residual functional compounds remain after primary processing and extraction.	[19,29,30]
Buckwheat plants and hulls	Rutin and other flavonoids	Underutilized edible parts (leaves, stems, and flowers) contain higher flavonoid levels than the commonly consumed grain.	[31,32]

residues, citrus peels and pomace, onion skins, sesame or perilla residues, and ginseng marc. Although generated during routine food processing, these materials retain measurable levels of phenolics, flavonoids, isoflavones, lignans, sterols, polysaccharides, and related functional compounds.

Rice bran is one of the most extensively studied cereal byproducts in Korea. Ethanol extracts showed higher total polyphenol, total flavonoid, γ-oryzanol, vitamin E, and antioxidant activity than half-crushed rice. Alkaline hydrolysis followed by cleanup further enriched phenolic acids, including ferulic acid and sinapic acid.[20,21]

Soybean processing residues represent another major matrix group. A comparative study of dehulled meal, feed meal, soybean hull, heat-treated hull, soybean curd residue, soybean curd whey, and soaking water demonstrated that isoflavone contents varied substantially according to processing route and moisture condition.[22]

Citrus byproducts are represented mainly by *Citrus unshiu* peel and pomace. Pomace extracts were evaluated using antioxidant-related indices, whereas peel studies focused on specific flavonoids such as hesperidin and narirutin.[23-25] Methoxylated and glycosylated citrus flavonoids have also been applied as practical marker compounds in analytical investigations of citrus matrices.[17,18]

Onion byproducts are primarily represented by skin and outer layers. Quercetin concentration was highest in the skin, followed by the outer, middle, and core tissues, and this distribution pattern was consistent with DPPH radical-scavenging activity.[26]

Oilseed byproducts include sesame dregs and defatted perilla seed residue. Sesame dregs retained β-sitosterol, daucosterol, sesamin, and sesamolin after chromatographic separation. Defatted perilla residue remained a protein- and phenolic-containing matrix responsive to enzymatic treatment.[27,28]

Ginseng byproducts constitute another representative Korean category. Ginseng marc retained soluble acidic polysaccharides, whereas red ginseng marc contained less-polar metabolites and showed compositional differences across solvent fractions.[19,29,30]

In addition to processing residues, several edible but underutilized plant parts have also been examined. In buckwheat, stems, leaves, and flowers are edible but are not commonly consumed. HPLC analysis showed that these fractions contained higher rutin levels than the edible grain. Buckwheat hulls were likewise reported to contain flavonoids, including rutin, quercetin, orientin, and vitexin.[31,32]

2.2. Major target compounds

The reviewed Korean studies show that analytical targets in agri-food byproducts can be grouped into several practical classes. The first class comprises phenolic acids and flavonoids. Representative compounds include ferulic acid and related phenolic acids in rice bran, hesperidin and narirutin in citrus residues, quercetin derivatives in onion byproducts, and rutin and other flavonoids in buckwheat plants and hulls.[20,21,23-26,31-33] These compounds are frequently selected as markers because they are relatively abundant, characteristic of specific matrices, and commonly linked to antioxidant screening data.

The second class is isoflavones, which are dominant in soybean processing residues. Typical analytical targets include daidzein, glycitein, genistein, and their conjugated precursors following hydrolysis.[22]

The third class includes lipophilic antioxidants and lignan-related compounds. Rice milling byproducts contain γ-oryzanol and vitamin E in addition to phenolics, whereas sesame dregs contain sesamin, sesamolin, and phytosterols.[20,27] These findings indicate that some Korean byproducts require analytical attention to both polar and non-polar fractions rather than a phenolics-focused approach.

The fourth class comprises polysaccharides, ginsenosides, and other structurally complex metabolites. Studies on ginseng byproducts have emphasized soluble acidic polysaccharides, ginsenosides, and less-polar metabolites remaining after processing.[19,29,30]

Overall, analytical targets in Korean agri-food byproducts range from single-marker small molecules to broader compositional fractions. Target selection should therefore reflect the original chemical characteristics of each matrix rather than rely on a uniform cross-matrix strategy. This matrix-dependent diversity

provides the basis for the pretreatment, extraction, and instrumental considerations discussed in the following section.

3. Analytical Approaches and Representative Applications

The analysis of bioactive compounds in Korean agri-food byproducts generally involves four steps: sample pretreatment, extraction or hydrolysis, instrumental analysis, and data interpretation (*Table* 2). Previous reviews indicate that extraction efficiency is primarily influenced by solvent polarity, temperature, extraction time, particle size, and matrix interaction.[1-3] Selection of analytical methods depends on analyte polarity, volatility, concentration level, and the degree of structural confirmation required. Across the reviewed studies, conventional solvent extraction and acid or alkaline hydrolysis are commonly applied as preparative steps. Targeted chromatographic analysis remains the principal approach, whereas LC–MS/MS, GC–O–MS, and NIR-based screening are applied in more specific contexts.[4-10,14] These platforms serve different analytical purposes. Screening assays and IR/NIR tools improve throughput and support relative comparison, but they are limited in selectivity and require calibration against reference methods for quantitative use. Targeted LC methods provide compound-specific quantification with higher selectivity, but performance depends on matrix effects, calibration strategy, and validation design. GC-based workflows remain essential for volatile and lipid-derived fractions, particularly when aroma or oxidation markers are of interest. A tiered workflow can therefore be practical, with rapid screening used to prioritize samples or conditions followed by chromatographic confirmation for reporting and comparison.

3.1. Sample pretreatment and extraction

Sample pretreatment is inherently matrix-dependent

Table 2. Main analytical approaches used for Korean agri-food byproducts and their practical considerations

Approach	Main purpose	Representative methods	Main practical issue	Ref.
Pretreatment and extraction	Release or enrich target compounds before measurement	Drying, defatting, hydrolysis, solvent extraction, response-surface optimization	Conditions are matrix-dependent; comparability improves with clear reporting of key pretreatment/extraction variables and matrix-aware validation.	[21-24,33,34]
LC-based analysis	Targeted quantification of non-volatile compounds	HPLC-UV, HPLC-PDA, UHPLC, LC-MS/MS	Selectivity and matrix effects become more important as methods move from simple markers to multi-compound profiling.	[4-6,22,31,32,38-43,58]
GC-based analysis	Profiling of volatiles and aroma-active compounds	GC-MS, GC-O, HS-SPME-GC/MS, SDE-GC/MS	Major chromatographic peaks do not necessarily define sensory impact.	[7-10,46-53]
Spectrophotometric screening	Rapid comparison of extracts or conditions	DPPH, ABTS, reducing power, Folin-Ciocalteu	Results are global indices with limited specificity and can be influenced by matrix components; quantitative use requires calibration and external validation against chromatographic reference data.	[11-13,20,59]
IR/NIR screening	High-throughput estimation and classification	FTIR, NIR with chemometrics	Model performance depends on representative calibration sets and external validation; model transfer and periodic recalibration are often required when matrices or seasons vary.	[14-16]
Validation	Demonstration of method suitability	Specificity/selectivity, linearity, LOD/LOQ, recovery, precision, uncertainty	Validation should be fit-for-purpose and matrix-aware, not generic.	[38,41-43,56-58]

because target compounds differ in physical form and stability. General reviews on food phenolics and agri-food byproducts consistently report that extraction efficiency is influenced by solvent composition, temperature, extraction time, particle size, and matrix characteristics.[1-3] These factors are particularly relevant for plant residues in which phenolics may occur as free, bound, glycosylated, or otherwise conjugated forms.[2,3]

Across the reviewed studies, practical matrix-specific pretreatment and extraction strategies remain more common than highly intensified extraction systems. In rice bran, alkaline hydrolysis followed by ethyl acetate extraction yielded higher total polyphenol content and antioxidant activity than direct extraction, and C18 cleanup enriched ferulic acid and sinapic acid.[21] In soybean byproducts, acid hydrolysis under reflux was selected to convert isoflavone conjugates prior to HPLC analysis.[22] In citrus peel, response surface methodology was applied to optimize hesperidin extraction, and subcritical water extraction combined with pulsed electric field treatment improved recovery of hesperidin and narirutin from waste peel.[23,24] For onion peel, solvent concentration, temperature, pH, and solvent-to-solid ratio were optimized using response surface methodology.33 Similar matrix-specific optimization was reported in a related Korean study on HPLC–UV analysis of six active compounds in *Orostachys japonicus*.[34] From a sustainability perspective, extraction workflows are increasingly discussed under green analytical chemistry principles, which aim to reduce solvent hazard, energy demand, and waste generation while maintaining data quality. In byproduct analysis, practical options include using aqueous ethanol instead of more hazardous organic solvents, applying assisted extraction (e.g., ultrasound- or microwave-assisted extraction) to reduce time and energy input, and using pressurized or subcritical water extraction when targets are sufficiently stable. Deep eutectic solvents have also been explored for selected byproduct matrices, but method development should include checks for analyte degradation, co-extraction of interferents, and changes in matrix effects that affect downstream chromatography.[35-37]

In this context, standardization is best approached through consistent reporting of key pretreatment variables and matrix-aware validation, rather than through a single protocol applied across different byproduct matrices. This consideration is particularly important in Korean byproduct research, where rice bran, citrus peel, onion skin, oilseed residues, and ginseng marc differ substantially in polarity profile, moisture content, and analyte state.

3.2. LC-based methods for non-volatile compounds

LC-based methods remain the primary analytical platforms for non-volatile compounds in Korean agri-food byproducts. General chromatographic reviews indicate that HPLC with UV or PDA detection is suitable for routine targeted analysis, whereas UHPLC and LC–MS/MS provide higher sensitivity, improved separation efficiency, and stronger structural confirmation in complex matrices.[4-6] These advantages are particularly relevant for phenolic acids, flavonoids, isoflavones, lignans, and ginsenoside-related compounds that frequently co-occur with interfering matrix components.

The reviewed application studies are consistent with this pattern. Soybean isoflavones were quantified after acid hydrolysis by HPLC–UV.[22] Flavonoid markers in onion extracts and in buckwheat aerial parts and hulls were quantified by HPLC–PDA, with retention time and PDA spectral agreement supporting specificity.[31,32,38] Citrus flavonoids have been analyzed by targeted LC methods using methoxylated and glycosylated derivatives or hesperidin and narirutin as practical markers.[17,18,23,24] Ginsenosides and related metabolites in ginseng-derived matrices have also been characterized by targeted LC approaches.[19,29]

A validated HPLC–PDA procedure has also been reported for marker quantification in Korean byproduct matrices, such as γ-oryzanol determination in fermented rice bran.[39] An HPLC–DAD method was also optimized and validated for adenosine and cordycepin in cordyceps products.[40] For sesame-derived lignans, Jung *et al*. validated an HPLC–PDA method for sesamol, sesamin,

and sesamolin in fermented sesame, whereas Kim *et al.* developed an LC–MS/MS method for five lignans.[41,42] The latter method showed good selectivity, linear response, low detection limits, recoveries of 90.6–109.1 %, and repeatability and reproducibility below 5 %.[42] Seo *et al.* further demonstrated the value of matrix-matched calibration in HPLC–UV analysis of quercetin across different food matrices.[43] Overall, LC-based methods remain the first-line choice for non-volatile bioactive compounds in Korean byproducts, but matrix-aware validation becomes increasingly important as analytical scope expands from single-marker quantification to multi-compound profiling. Beyond targeted quantification, non-targeted LC–HRMS metabolomics can be used to assess broader chemical fingerprints, support marker discovery, and compare batches or processing conditions in complex byproduct extracts. For fit-for-purpose use, this approach requires a defined QC strategy (e.g., pooled QC samples and feature filtering), transparent reporting of data processing steps, and explicit confidence levels for compound annotation. In practice, non-targeted profiling is most informative when key features are confirmed by MS/MS and, where possible, by authentic standards.[44-45]

3.3. GC-based methods for volatile and lipophilic compounds

GC-based methods are primarily applied to volatile and aroma-active compounds in the reviewed Korean literature. Recent methodological reviews describe four main steps in volatile analysis: isolation and concentration, chromatographic separation, compound identification, and sensory evaluation.[7-10] GC–MS remains the principal instrumental platform, while SPME, SDE, SAFE, and GC–O–MS are selected according to matrix characteristics and study objectives.[7-10]

Korean citrus studies provide representative examples. Cold-pressed peel oils of Satsuma mandarin, Shiranui, and Kiyomi were profiled by GC or GC–MS. Limonene was consistently the dominant component, but differences in aldehydes, alcohols, esters, and sesquiterpenes were observed across cultivars and geographic origins.[46-49] Jeju citrus varieties, as well as yuzu, kumquat, lemon, and lime, were analyzed by simultaneous distillation–extraction followed by GC–MS, demonstrating distinct cultivar-dependent volatile profiles.[49,50]

GC–olfactometry added a sensory dimension to compositional analysis. In Hallabong peel oil, citronellal, cis-β-farnesene, and citronellyl acetate were identified as key odor-active compounds despite the predominance of limonene.[51] Citrus peel essential oils characterized by hydrodistillation and GC–MS have also been associated with antimicrobial activity.[52] In addition, HS-SPME–GC/MS has been applied as a practical approach for fruit volatiles, including Fuji apple, and headspace-based methods have been reviewed from a methodological perspective.[8,53] Beyond citrus peel oils, GC-based workflows have also been applied to other byproduct matrices for headspace monitoring or characterization of lipid-derived fractions. For rice bran and rice bran oil, headspace oxygen content has been measured by GC–TCD to assess oxidative stability under heat or light exposure, supporting evaluation of stabilization and storage conditions.[54] For ginseng processing residues, red ginseng marc oil obtained from the post-extraction byproduct has been chemically characterized using GC-based profiling, providing a non-citrus example of byproduct-derived lipid fraction analysis.[55] These examples indicate that GC platforms can address both aroma-related questions and lipid-associated markers, depending on matrix type and study purpose.

Overall, GC-based analysis in the reviewed Korean byproduct literature has focused mainly on citrus peel volatiles, but it has also been used for headspace oxygen monitoring and characterization of lipid-derived fractions in other matrices.

3.4. Spectrophotometric and spectroscopic screening

Spectrophotometric assays remain practical tools for rapid screening of Korean agri-food byproducts. DPPH, ABTS, reducing power, and Folin–Ciocalteu assays are simple and cost-effective, and they are suitable for preliminary comparison of cultivars, extracts, and processing conditions.[11,20,25,26] In rice

milling byproducts, for example, DPPH, ABTS, reducing power, total polyphenol, total flavonoid, γ-oryzanol, and vitamin E measurements were combined to compare bran and half-crushed rice extracts.[20] However, methodological reviews have noted clear limitations. The Folin–Ciocalteu reaction is based on electron transfer and responds to non-phenolic reducing substances, making matrix effects and experimental conditions critical determinants of the result.[12,13] These assays should therefore be interpreted as global screening indices rather than compound-specific alternatives to chromatographic methods.[11-13]

Infrared (IR) and near-infrared (NIR) spectroscopy provide complementary rapid screening approaches. These techniques are fast and non-destructive, but analytical performance depends on analyte class, matrix characteristics, wavelength selection, spectral preprocessing, and calibration design.[14] NIR applications require representative calibration sets, appropriate chemometric modeling, and consideration of postharvest and seasonal variation.[16] In a Korean sesame study, NIR was applied to estimate sesamin and sesamolin contents for breeding purposes, with stronger predictive performance for sesamin.[15] When spectroscopy is used for monitoring, the model should be built against reference LC/GC results, tested using an independent validation set, and updated when matrix or seasonal variability affects prediction performance.

Overall, spectrophotometric assays and spectroscopic methods can support rapid screening and trend monitoring when they are calibrated and externally validated against chromatographic reference data, but they do not replace compound-specific identification and quantification.

3.5. Validation and practical considerations

Validation should be considered a fit-for-purpose process rather than a formal add-on to instrumental analysis. The AOAC single-laboratory guideline emphasizes applicability, selectivity, calibration, accuracy, repeatability, intermediate precision, and limits of detection and quantification, while the ICH Q2(R2) framework similarly highlights specificity or selectivity, working range, accuracy, precision, and robustness.[56,57] These principles are directly relevant to agri-food byproduct analysis, where extraction conditions, hydrolysis steps, and matrix composition can influence analytical performance.

The reviewed Korean validation studies provide practical examples. Quercetin in onion extract was validated by HPLC–PDA with acceptable specificity, linearity, recovery, and intra- and inter-day precision.[38] Sesame lignans were validated by HPLC–PDA in fermented sesame and by LC–MS/MS in edible seed matrices, demonstrating high linearity, recoveries near 100 %, and repeatability or reproducibility below 5 %.[41,42] Quercetin quantification across different food matrices was further improved by matrix-matched calibration and explicit reporting of measurement uncertainty.[43]

Validation studies outside the Korean byproduct field support similar conclusions. Simultaneous HPLC–DAD analysis of multiple phenolics can achieve acceptable linearity, sensitivity, recovery, and repeatability when experimental conditions are controlled.[58] For screening assays, microplate-based Folin–Ciocalteu and DPPH methods can achieve analytical equivalence to conventional procedures within defined working ranges.[59]

Overall, current evidence indicates that Korean byproduct analysis is progressing beyond simple marker quantification. However, clearer matrix-specific validation and more consistent reporting remain necessary, particularly when chromatographic data are used to support functional ingredient development or to calibrate rapid spectroscopic methods. For practical comparability, studies should report a minimum set of analytical metadata, including byproduct origin and processing history, storage and thermal history, moisture basis and units (FW or DW), key pretreatment parameters (drying/inactivation, particle size, defatting, hydrolysis), extraction conditions (solvent composition, ratio, time, temperature), calibration strategy (internal standard and matrix-matched calibration when needed), and core validation metrics (LOD/LOQ, recovery, precision, and matrix effects).

4. Conclusions and Future Perspectives

Korean agri-food byproducts have been reported as sources of phenolic acids, flavonoids, isoflavones, lignans, ginsenoside-related compounds, polysaccharides, and volatile constituents. Across the studies reviewed here, analytical workflows most commonly combine matrix-specific pretreatment with targeted LC-based quantification of non-volatile compounds and more selective GC-based analysis of volatiles.

The reviewed literature also indicates that spectrophotometric assays and IR or NIR methods are used mainly for rapid screening. Their performance is typically calibration-dependent and less selective than chromatographic platforms. These approaches are most useful as complementary tools for comparative screening and trend monitoring when calibrated and validated against chromatographic reference methods, rather than as stand-alone substitutes for compound-specific analysis.

Across the surveyed studies, reporting of key pretreatment variables and validation metrics is uneven, which limits direct comparison of absolute concentrations across matrices and laboratories. More consistent documentation of pretreatment conditions and core validation results would help interpret differences across studies more reliably. Overall, the studies reviewed here suggest that more consistent pretreatment, matrix-aware validation, and clear reporting may improve comparability across studies. This review does not aim to cover analytical methods in general. Instead, it compiles how commonly used workflows have been applied to representative Korean agri-food byproduct matrices. It summarizes matrix-specific choices and practical points for pretreatment, extraction, chromatographic analysis, rapid screening, and validation. Because the reviewed literature represents a selected set of published studies, this summary may not reflect all Korean byproduct research. Within this limitation, the framework presented here may serve as a practical guide for designing and interpreting analytical studies on Korean agri-food byproduct utilization.

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