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COLD PRESSING, AMYGDALIN REMOVAL AND CLOSED-CYCLE VALORISATION OF STONE FRUIT KERNEL OILS: A MASS-BALANCE ASSESSMENT

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Abstract:

Stone fruit processing in Uzbekistan generates a large kernel stream that is currently burnt or landfilled, although the kernel contains oil at a level comparable with conventional oilseeds. Three recovery routes petroleum-fraction extraction, hot pressing and cold pressing were compared on six kernel feedstocks, the sanitary and ecotoxicological status of the cold-pressed oils was evaluated, and a water-free route for removing amygdalin with closed-loop recovery of the extractant is reported. Extraction gave the highest yields (40.1–48.2 %), but cold pressing retained on average 77.7 % of the extraction yield while eliminating solvent residues and volatile organic emissions. The 22.3 % apparent shortfall is not a loss: it is recovered in the press cake, so that about 101 kg of oil per 1000 kg of kernel is exchanged for roughly 91 kg of additional cake. Amygdalin ranged from 0.0936 mg/kg in plum to 0.430 mg/kg in peach, a 4.6-fold spread across species grown on the same soils, and reached 0.761 mg/kg in the almond group, 1.52 times the 0.5 mg/kg limit. Toxic metals occupied 0.2–24.5 % of their permitted maxima, aerobic counts stayed below 10 CFU/g, and all indicators remained within specification over two years of ambient storage. Ethanolic extraction (96 % ethanol, oil : ethanol 1:1 v/v, 30–40 °C, 40 min) recovered amygdalin as a crystalline solid and returned the ethanol to the cycle. The mass balance on 1000 kg of dry kernel closes with 335 kg of purified oil, 655 kg of cake and 0.5 kg of amygdalin, 99.05 % of the feedstock entering a marketable or secondary stream. Because amygdalin content is species-determined rather than site-determined, targeting the purification step by species is proposed as the governing design principle.

Keywords: stone fruit kernel oil, cold pressing, amygdalin, cyanogenic glycoside, ethanolic extraction, press cake valorisation, mass balance, waste-free processing.

Introduction

Fruit processing occupies an awkward position in the resource economy of Uzbekistan. Apricot, peach,

plum, sour cherry and almond are grown at scale, yet the kernel left after pulping is treated as a disposal problem: at the fruit-processing plants of Navoi region it is burnt for heat or sent to landfill. The kernel is not a poor material. Reported oil contents overlap those of conventional oilseeds, and the oils are rich in oleic and linoleic acids, tocopherols and phytosterols [1].

What the kernel carries, and conventional oilseeds do not, is a cyanogenic glycoside. Amygdalin hydrolyses under emulsin to glucose, benzaldehyde and hydrogen cyanide, so its residual concentration in the oil, rather than the yield of the pressing step, sets the ceiling on what the material may be used for. Bolarinwa et al. [2] found amygdalin concentrations spanning several orders of magnitude across commercial seeds and kernels, and Makovi et al. [3] reported a comparably wide spread between bitter and sweet apricot kernels. The European Food Safety Authority set an acute reference dose of 20 µg CN per kg of body weight for raw apricot kernels [4] and extended the assessment to other foods [5]; the toxicology and the analytical options have been reviewed in detail [6], [7].

The regulatory picture is not consistent. Commission Regulation (EU) 2023/915 sets maximum hydrocyanic acid levels for apricot kernels and related products [8], whereas CODEX STAN 193-1995 carries no corresponding indicator [9], and the Regulation exempts seeds crushed for oil production and refining an exemption that leaves cold-pressed, unrefined kernel oils in a methodological gap. Cai et al. [10] showed that cyanogenic glycosides do pass into cold-pressed oil and can be quantified there at trace level, which is precisely the case the exemption does not cover.

Two routes out of this constraint are described. The first is to choose the recovery method so that less glycoside is carried over: cold pressing has been compared with supercritical carbon dioxide on apricot kernel oil, supercritical CO_2, hexane extraction and cold pressing have been set side by side on six species, and pressing has been compared with ultrasound-assisted and Soxhlet extraction. The second is to destroy the glycoside after recovery, by ultrasound-accelerated debittering [11] or by lactic acid bacteria [12]. Both operate in aqueous media, which is the difficulty: water is the reagent of the hydrolysis that liberates hydrogen cyanide.

The kernel stream also has a second, larger fraction: between 60 and 70 % of the kernel mass leaves the press as cake, and its fate decides whether the process is waste-free in any meaningful sense. Cold-pressed oilseed cakes have been reviewed as feed ingredients press cakes and meals as circular-economy feedstocks, and apricot kernel cake as a source of bio-functional protein and of nutritionally valuable by-products [13], [14].

The literature therefore establishes that kernel oils are chemically attractive, that amygdalin is the binding constraint, and that the cake is a resource rather than a residue. What it does not provide is a route that removes amygdalin from the oil without water, recovers the extractant, converts the removed glycoside into a product, and closes a mass balance over the whole kernel. No systematic work of this kind has been reported for Uzbek raw material, where the oils-and-fats literature is dominated by cottonseed oil, soapstock and gossypol resin. The present paper addresses that gap, continuing the authors' earlier characterisation of these oils and of the defatted cake [15], [16].

Materials and Methods

Kernels of peach, plum, sour cherry, apricot, bitter almond and wild almond were collected from the Khatirchi and Nurata districts of Navoi region. All six feedstocks were grown on the same soil-climate zone, which is what makes the between-species comparison of amygdalin meaningful. Kernels were

separated from the shell and dried to a residual moisture below 5 %. Three recovery routes were run in parallel on each feedstock in three replicates extraction with a petroleum fraction, hot pressing, and cold pressing at room temperature and yield is expressed on the absolutely dry kernel mass. All results reported after Section 3.1 refer to cold-pressed oils.

Amygdalin was determined by high-performance liquid chromatography on an LC-2030C 3D Plus instrument with a Shim-pack GIST-HP C18 column (250 × 4.6 mm, 5 µm); mobile phase acetonitrile : water (55:45) at 0.5 mL/min, column temperature 40 °C, detection at 215 nm, quantification against a commercial reference standard. Mercury, lead, cadmium, arsenic, copper and iron were determined by inductively coupled plasma mass spectrometry on an AT 7500 instrument at 1200 W plasma power and referred to the permitted maxima of the governing specification. Total aerobic and fungal counts, Escherichia coli, Enterobacteriaceae and Salmonella were assessed by the agar method.

Ethanol of chemical purity (96 %) was chosen as the extractant for amygdalin removal rather than water, because in the presence of water, and particularly under emulsin, amygdalin decomposes according to $C_{20}H_{27}NO_{11} + 2H_2O \rightarrow 2C_6H_{12}O_6 + C_6H_5CHO + HCN$, and both benzaldehyde and hydrogen cyanide are toxic; a purification step carried out in water would contaminate the oil it was meant to clean. The pressed oil was filtered and mixed with 96 % ethanol at 1:1 by volume, extracted for 40 min at 30–40 °C under mechanical stirring and transferred to a settling column; after phase separation the purified oil was drawn off through the lower valve, while the ethanolic phase was evaporated, condensed through a cooler and returned to the cycle, and the solid recovered from it was collected.

Storage stability was established by natural observation rather than by an accelerated test: samples were held in 50 and 100 mL amber plastic containers at 10–25 °C for two years, and acid value, saponification value, iodine value and density were recorded initially and after 6, 12, 18 and 24 months. The mass balance of Section 3.5 was calculated on 1000 kg of absolutely dry kernel using the mean cold-pressing yield and the fractions measured here.

Results and Discussion

The three routes do not rank in the same order for every feedstock (Fig. 1). In peach, plum and sour cherry the yield falls monotonically from extraction through hot pressing to cold pressing, from 41.8 to 39.6 and 33.5 % in peach and from 41.6 to 33.5 and 28.6 % in sour cherry. In apricot and bitter almond hot pressing catches up with extraction or overtakes it: apricot gives 48.0 ± 0.5 % on hot pressing against 46.3 ± 0.2 % on extraction.

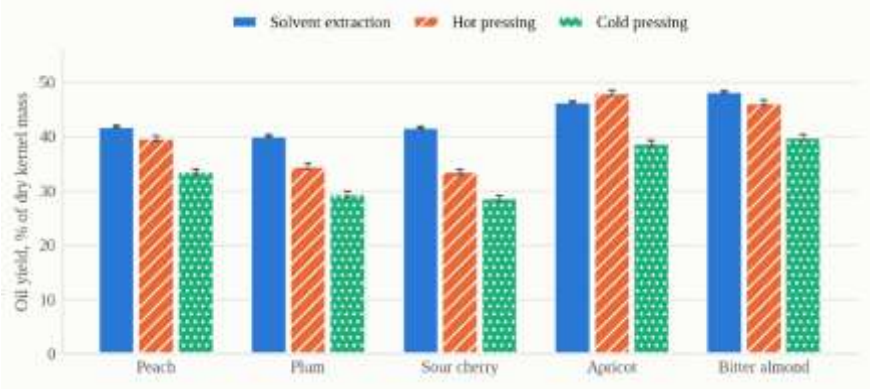

Fig. 1. Oil yield from five kernel feedstocks by solvent extraction, hot pressing and cold pressing. Error bars are the standard deviation of three replicates.

Yield alone is not a sufficient criterion once the comparison is made on environmental terms. Solvent extraction consumes a petroleum fraction per kilogram of feedstock, part of which remains in the product as a residue and part of which leaves the plant as a volatile organic emission requiring recovery; the regulatory pressure on hexane and the terms of its replacement have been set out by Carré et al. Cold pressing runs at room temperature with no solvent and no added heat: the yield is 5 to 13 percentage points lower, but no solvent residue is carried over and the thermolabile fraction survives, which is what makes cold-pressed kernel oils attractive in the first place.

The size of the penalty is worth stating precisely, because it is the usual argument against the solvent-free route. Taking the extraction yield as 100 %, cold pressing retains 83.6 % in apricot, 82.6 % in bitter almond, 80.1 % in peach, 73.3 % in plum and 68.8 % in sour cherry, a mean of 77.7 %. The remaining 22.3 % is not destroyed: it stays in the press cake, and on 1000 kg of dry kernel about 101 kg of oil is exchanged for roughly 91 kg of additional cake. Whether that is a loss depends on what the cake is worth, and the literature is unambiguous that it is worth a good deal cold-pressed cakes are established feed ingredients, apricot kernel cake is a documented source of bio-functional protein, and press cakes generally are treated as circular-economy feedstocks. The yield argument for solvent extraction therefore does not survive a mass balance drawn over the whole kernel.

Amygdalin in the cold-pressed oils spans a far wider range than the oils themselves suggest (Fig. 2). Plum oil contained 0.0936 mg/kg, bitter apricot oil 0.111 mg/kg and peach oil 0.430 mg/kg, a 4.6-fold difference between the extremes. All three are below the permitted maximum of 0.5 mg/kg, but the safety margin is not comparable: plum occupies 18.7 % of the limit and peach 86 %. Across the full set of cold-pressed samples the range was 0.111–0.761 mg/kg, the upper value belonging to the bitter and wild almond oils and exceeding the limit by a factor of 1.52.

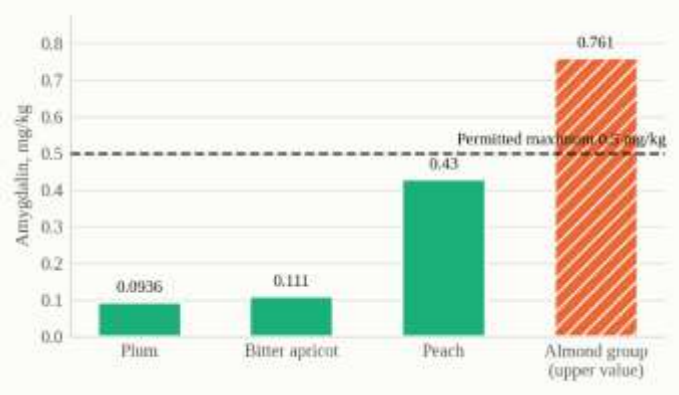

Fig. 2. Amygdalin content of the cold-pressed oils against the permitted maximum. The almond group is shown at its upper measured value.

The pattern carries a design consequence. All samples were grown in the same two districts, on the same soil-climate zone, so the 4.6-fold spread cannot be attributed to site. It follows the botanical species the genetically determined activity of the cyanogenic glycoside pathway in the seed and the same species-level control appears in the data of Bolarinwa et al. and Makovi et al. If amygdalin were site-determined, purification would have to be applied to the whole stream; because it is species-determined, it can be targeted: the almond group requires it unconditionally and peach because its margin is too narrow to rely on, while plum and bitter apricot do not. Oils too high in amygdalin are

not thereby waste they remain usable in pharmaceutical and cosmetic applications, where the product does not enter the gastrointestinal tract and the enzymatic hydrolysis has no opportunity to proceed.

Vegetable oils are a sensitive indicator of technogenic contamination, since the root system takes up mobile metal species from the soil and part of that load reaches the seed lipid fraction. All four toxic elements stayed well clear of their limits (Fig. 3). Cadmium and arsenic occupied less than 1 % of the permitted level, cadmium at 0.0001–0.0002 mg/kg against a 0.05 mg/kg limit and arsenic at 0.0002–0.00084 mg/kg against 0.1 mg/kg. Mercury showed the highest relative occupancy at 18.0–24.5 %, the maximum belonging to peach oil, and lead followed at 11.7–16.9 %. These are the elements that accumulate near industrial plant and transport corridors; their near-absence is consistent with the low technogenic load of the mountain and foothill zones of Khatirchi and Nurata, and the values sit at the low end of the range compiled for edible vegetable oils by González-Torres et al.

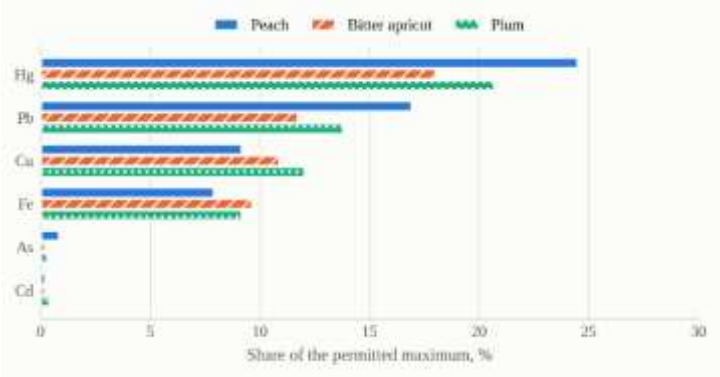

Fig. 3. Toxic and catalytically active metals in the cold-pressed oils, expressed as a share of the permitted maximum for each element.

Iron and copper matter for a different reason: both catalyse the initiation step of lipid peroxidation, so their concentration predicts shelf life rather than toxicity. Iron occupied 7.9–9.6 % of its limit and copper 9.2–12.0 %, an order of magnitude below the level at which chain oxidation is accelerated. Microbiologically the oils were effectively sterile: aerobic and fungal counts were below 10 CFU/g, at least three orders of magnitude below the limits of 10^4 and 2×10^2, and no pathogens were detected. The kernel is naturally isolated inside a hard shell, and the lipid phase is a hostile medium of low water activity, so cells introduced incidentally during pressing cannot multiply; Drewnowska and Swiecicka reached a comparable conclusion for cold-pressed oils.

These two observations converge in the storage behaviour. Over 24 months at 10–25 °C the acid value rose from 0.41 to 0.43 mg KOH/g in peach oil (4.9 %) and by 2.6 % in bitter apricot and plum oils, every value remaining more than a factor of five below the 2.0 mg KOH/g limit. Iodine value was effectively unchanged 100.84 throughout in bitter apricot oil, 99.9 to 100 in peach oil so the double bonds were not consumed and the autoxidation chain did not start; density rose by 0.001 g/cm³ in the second year, at the edge of measurement error; and in peach oil linoleic and oleic acids moved from 16.51 and 74.99 % to 16.50 and 74.80 %. A two-year shelf life without antioxidant addition follows directly from the low catalyst content, consistent with the oxidative behaviour reported for these oils under thermal and photo-oxidative stress and for plum seed oil.

The solid recovered from the ethanolic extract was a white crystalline material, freely soluble in water, whose solution carried the characteristic bitter-almond odour a qualitative confirmation that the separated fraction is amygdalin. Three features of the route matter for its environmental accounting.

The ethanol is recovered at the evaporation and condensation stage and returned to the cycle, so the process runs in a closed loop and generates no liquid effluent, in contrast with the petroleum fraction of the extraction route. The separated amygdalin is not discarded but collected in crystalline form, where it serves as a pharmaceutical reference substance and feedstock. And the route brings into economic circulation a kernel resource that is at present burnt or landfilled.

The mass balance on 1000 kg of absolutely dry kernel closes as shown in Fig. 4: purified oil accounts for 335 kg (33.5 %), press cake for 655 kg (65.5 %), crystalline amygdalin for 0.5 kg (0.05 %) and process losses mechanical losses at the pressing, filtration and phase-separation stages for 9.5 kg (0.95 %). Recovered ethanol re-enters the cycle and does not appear as an output. In total 99.05 % of the feedstock enters a marketable or secondary stream and no landfill stream is formed.

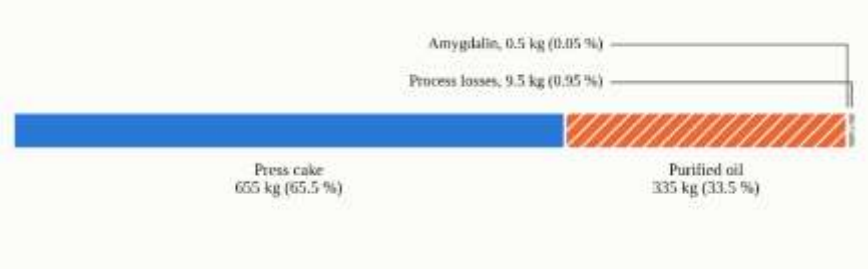

Fig. 4. Mass balance of integrated kernel processing, per 1000 kg of absolutely dry kernel. Recovered ethanol returns to the cycle and is not shown as an output.

The comparison with the conventional route is instructive in both directions. A solvent extraction flowsheet introduces several hundred kilograms of organic solvent into circulation per tonne of oil, a fraction of which leaves the system through evaporation, uncaptured emission and product residue; here the corresponding stream is ethanol, it is recovered, and the only genuinely lost material is the 0.95 % of mechanical losses. More striking is what happens to the toxic fraction: amygdalin, a hazardous separation product in the conventional accounting, appears here as a separate commodity stream, and that inversion is what makes the scheme waste-free in more than a nominal sense.

Conclusions

Of the three recovery routes compared on six kernel feedstocks, solvent extraction gave the highest yields (40.1–48.2 %) but is not acceptable on environmental grounds because of solvent residues in the product and volatile organic emissions from the plant. Cold pressing yields 5 to 13 percentage points less and retains a mean of 77.7 % of the extraction yield; the 22.3 % shortfall is transferred to the press cake rather than lost, which on 1000 kg of kernel exchanges about 101 kg of oil for roughly 91 kg of cake.

Amygdalin in the cold-pressed peach, bitter apricot and plum oils ranged from 0.0936 to 0.430 mg/kg, below the 0.5 mg/kg limit, and reached 0.761 mg/kg in the almond group, 1.52 times the limit. Because all samples came from the same soil-climate zone, the 4.6-fold spread is species-determined rather than site-determined, so the purification step should be routed by species: unconditionally for the almond group, on margin grounds for peach, and not at all for plum and bitter apricot.

All toxic metals occupied no more than 24.5 % of their permitted maxima and cadmium and arsenic less than 1 %; iron and copper, the oxidation catalysts, stayed below 12 %. Aerobic and fungal counts were below 10 CFU/g and no pathogens were detected. Over two years of ambient storage the acid

value rose by 2.6–4.9 %, the iodine value was unchanged and the fatty acid composition shifted by no more than 0.25 %, supporting a two-year shelf life without antioxidant addition.

A water-free ethanolic route (96 % ethanol, oil : ethanol 1:1 v/v, 30–40 °C, 40 min) removes amygdalin while avoiding the hydrolysis that liberates hydrogen cyanide, recovers the extractant in a closed loop and delivers the glycoside as a crystalline pharmaceutical feedstock. The mass balance closes at 335 kg of purified oil, 655 kg of cake and 0.5 kg of amygdalin per 1000 kg of dry kernel, with 0.95 % process losses, so 99.05 % of the feedstock enters a marketable or secondary stream. The yields, amygdalin concentrations, metal and microbiological values and storage data are taken from the experimental record; the retained-share percentages, the oil-to-cake exchange and the mass balance are calculated from those measurements.

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