

Fragrant Fakery: Sniffing Out the Truth in "Pure" Green Coffee Oil

Faux parfums: Flairer la vérité dans l'huile de café vert « pure »

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Abstract | Résumé

The natural extracts industry is plagued by imitation products that pose health risks, despite oversight by agencies such as the FDA and Health Canada. Coffee oils are a popular extract and are particularly susceptible to adulteration. The study compares the store-bought oil to a pure cold-pressed extract of green coffee beans using gas chromatography–mass spectrometry to find signs of adulteration. The store-bought oil exhibited markers of adulteration such as cyclamen aldehyde (cyclamal) and isopropyl myristate, while lacking several natural compounds such as palmitic acid and vitamin E found in cold-pressed green coffee oil (GCO). In comparison, through examination, the results show that an unregulated product, a “pre-workout” powder, is reported to contain methylhexanamine (DMAA) and its analogue DMHA, both of which were indeed identified, supporting the authenticity of the supplement. These results highlight the need for enforcement of regulations for consumer safety.

L'industrie des extraits naturels est rongée par des produits d'imitation qui présentent des risques pour la santé, malgré la surveillance d'agences telles que l'Agence américaine des produits alimentaires et médicamenteux (FDA) et Santé Canada. Les huiles de café sont un extrait populaire et sont particulièrement susceptibles d'être adultérées. L'étude compare l'huile achetée en magasin à un extrait pur pressé à froid de grains de café vert en utilisant la chromatographie en phase gazeuse couplée à la spectrométrie de masse pour détecter des signes d'adultération. L'huile achetée en magasin présentait des marqueurs d'adultération tels que l'aldéhyde de cyclamen (cyclamal) et le myristate d'isopropyle, tout en manquant de plusieurs composés naturels tels que l'acide palmitique et la vitamine E présents dans l'huile de café vert pressée à froid. En comparaison, à l'examen, les résultats montrent qu'un produit non réglementé, une poudre « pré-entraînement », contient de la méthylhexanamine (DMAA) et son analogue DMHA, tous deux identifiés, ce qui confirme l'authenticité du complément. Ces résultats soulignent la nécessité de faire respecter les réglementations pour la sécurité des consommateurs.

Keywords: green coffee oil; adulteration; GC-MS; natural product authentication; DMAA; DMHA; cosmetic chemistry; phytochemical analysis; consumer safety; synthetic additives

Introduction

Natural extracts are widely used commodities for scenting, skin hydration and flavouring. Valued at \$18.6 billion USD in 2020, it stands as a growing global industry (1). However, studies analyzing various natural oils have found that many commercially available “pure” products were adulterated with cheaper substitutes by exploiting legal loopholes, despite oversight enforced by the FDA and Health Canada (2,3). These adulterants pose documented potential health risks, including dermatological reactions such as rashes and psoriasis (4,5).

Green coffee oil (GCO) is challenging to extract as the raw beans have a tough, dense exterior and low oil yield. These physical characteristics make GCO particularly prone to adulteration as the process is labour-intensive and expensive (6, 7). GCO is used for skincare and fragrance because of its distinct aroma and natural

caffeine content. This study investigates the authenticity of commercially available GCO by comparing its chemical profile to that of pure, cold-pressed GCO using gas chromatography-mass spectrometry (GC-MS). As a parallel case, an unregulated athletic “pre-workout” supplement marketed as containing methylhexanamine (DMAA) is examined via acid-base extraction and GC-MS to determine its authenticity. DMAA is a prohibited stimulant for athletes in Canada and by World Anti-Doping Agency, but it is available in supplements (8). The purpose was to compare the ingredients of a “regulated” natural product (GCO) and an unregulated one (workout powder). Together, these analyses highlight the extent of adulteration in the natural products industry and the potential risks posed to consumers associated with long-term exposure to synthetic additives.

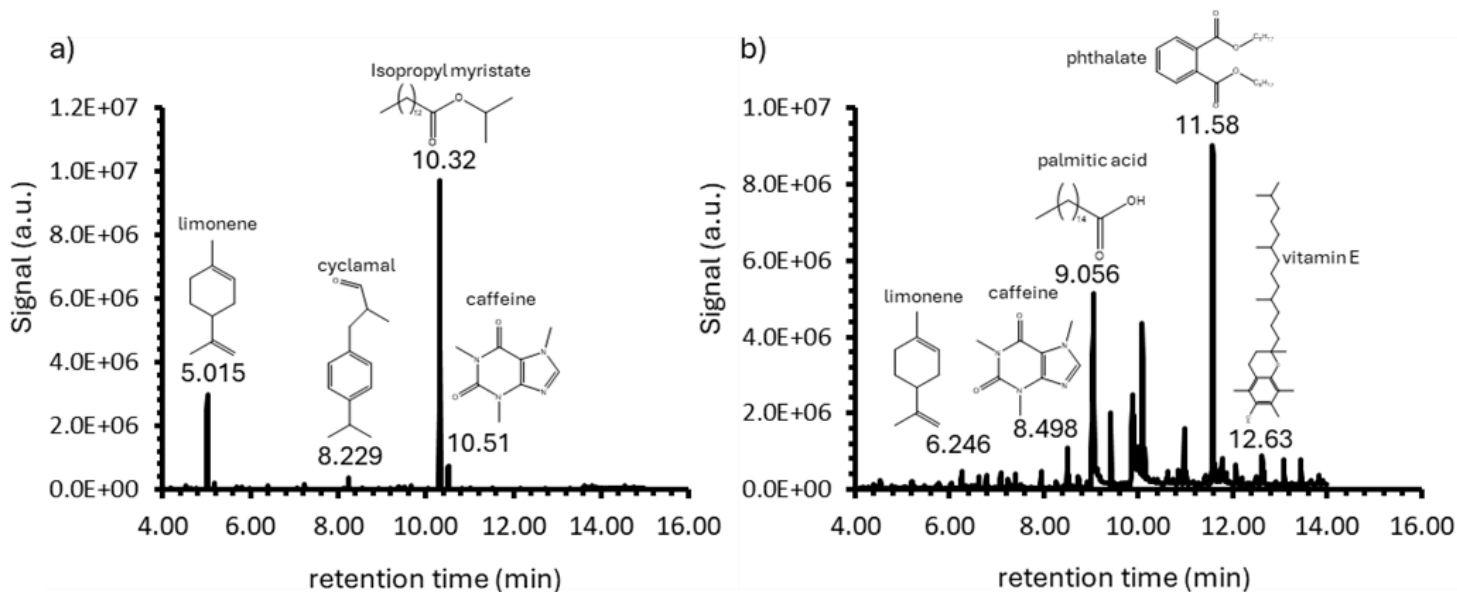


Figure 1. a) GC-MS results of store-bought GCO diluted in ethyl acetate. Peaks are labelled with respective compounds and their retention times. b) GC-MS results of cold-pressed GCO.

Experimental Procedures

Cold-Press

40 grams of green coffee beans were placed into the top of the cold-press. The entire nozzle was heated with a heat gun on the medium/low setting. The press was operated for 10 minutes or until oil is visible at the nozzle tip. Oil was extracted from the tip of the cold-press with a 10 μ L syringe and transferred to a vial.

Oil Analysis

1 μ L of store-bought or cold-pressed GCO was diluted to 100 μ L with ethyl acetate. 1 μ L of this solution was injected into the GC-MS using a DB-5 capillary column (split ratio 10:1, split flow: 12.8 mL/min, inlet temperature: 200 $^{\circ}$ C, oven program: 80 $^{\circ}$ C (2 min), ramp to 300-320 $^{\circ}$ C at 15-20 $^{\circ}$ C/min, carrier gas: Helium, detector: MS (electron ionization, 70 eV)). A solvent delay of four minutes was employed throughout. Compounds were identified by their retention index and mass spectrum.

Workout Powder Analysis

10 g of the pre-workout powder was dissolved in 250 mL of water and stirred for five minutes. The solution was acidified with 1 mol/L HCl until the pH was less than two. Liquid-liquid extraction was performed with hexane, retaining the aqueous layer. The aqueous layer was basified with six mol/L NaOH until the pH was greater than 11. Liquid-liquid extraction on this solution was performed with diethyl ether, retaining the top organic layer. One μ L of diethyl ether solution was diluted to 100 μ L in ethyl acetate. One μ L of this sample was injected into the GC-MS.

Results and Discussion

The chromatograms of commercial and cold-pressed GCO are shown in Figure 1. A visual comparison between Figures 1a and 1b reveals substantial differences in chemical composition. For example, the cold-pressed GCO contains peaks of several documented compounds in GCO, such as palmitic acid and vitamin E, both of which are very common in nature and plant oils in general (9-12). Limonene and caffeine were the only overlapping compounds, though only caffeine appeared at comparable abundance. The high abundance of phthalates comes from the packaging material used to store the green coffee beans, and can be a commonly found impurity in such products (13). In Figure 1a., the significant abundance of isopropyl myristate and cyclamal demonstrates that the store-bought oil was heavily adulterated, as these compounds are common emulsifiers and fixatives, respectively. Neither compound is naturally found in GCO.^{9,10} Cyclamal has been documented to cause dermatological reactions and is known to contribute to aquatic degradation (14). Isopropyl myristate causes rashes in prolonged usage and has various adverse symptoms when ingested (15, 16). The store-bought GCO also lacks palmitic acid and vitamin E found in cold-pressed GCO. Figure 2 confirms that DMAA and DMHA were both present in the pre-workout supplement, as reported on the ingredient list, supporting the authenticity of the product, despite the lack of regulation in the industry. This presents a stark contrast between the regulated natural extracts industry, hampered by imitation products and synthetic substitutions, and the unregulated pre-workout supplement industry. The adulterants found in the store-

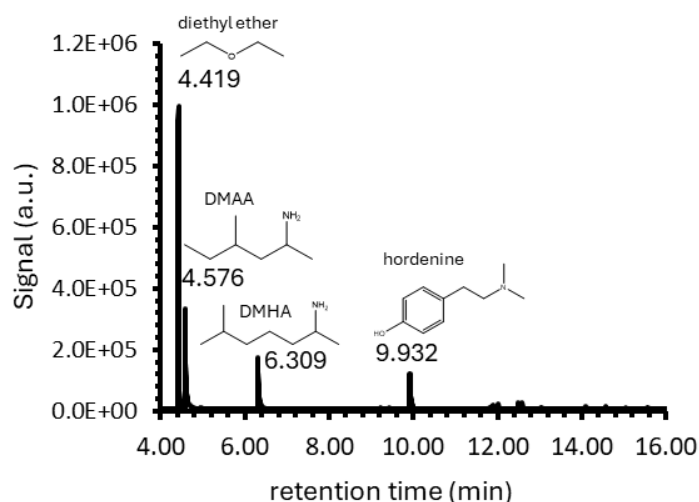


Figure 2. GC-MS results of extracted DMAA. Peaks are labelled with respective compounds and their retention times.

bought GCO, though relatively low in toxicity to humans, highlight the broader risk of substituting natural compounds with unregulated synthetic ones. Many deaths have been caused by this, as seen in the 2002 Slim10 Diet Pill incident and the 2009 Hydroxycut recall (17-19). As the industry grows rapidly, these findings demonstrate the need for tighter regulatory oversight and research into potential contraindications between adulterants and commonly used pharmaceuticals.

Conclusion

In this study, results show that a store-bought GCO was heavily adulterated compared to a cold-pressed GCO, despite current regulations and its marketing as a “pure” natural extract. The adulterants found may cause several different types of irritation. The pre-workout powder examined contains DMAA and its analogue, DMHA, as advertised, even without regulatory bodies overseeing the synthesis process. These results show that great strides in enforcement are required by regulatory bodies. The possibility of companies using toxic substituents, as has been seen in the past, alongside the growth of the industry, further emphasizes the need for regulation. Further research should be done into common adulterants and their contraindications with widespread medications to fully evaluate their safety.

Acknowledgements

The authors thank the JLH Mass Spectrometry Core Facility of the University of Ottawa for providing instrument access and consumables related to this project.

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