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The determination of unapproved drugs in New Zealand dietary supplements using validated chromatographic methods

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Abstract

In line with the increasing popularity of dietary supplements, adulteration of these products is becoming more common globally. In this study, validated LC and GC methods were used to test 68 dietary supplements found in the New Zealand online marketplace, spanning products advertised for sexual enhancement and weight loss. Of the sexual enhancement supplements, 11% of those tested contained an unapproved substance that was not listed on the product label, with the erectile dysfunction drug tadalafil and the prescription medicine yohimbine detected in these samples. For the product that contained tadalafil, a consumer taking the listed maximum serving size would consume more than double the maximum daily recommended dose for tadalafil (>40 mg). While no unapproved substances were detected in weight loss supplements, high caffeine levels were observed, with two weight loss supplements having listed maximum daily serving sizes that corresponded to greater than 400 mg caffeine. Caffeine was also detected in one sexual enhancement product that did not list caffeine or tea extract on its label. While these results represent a subset of the dietary supplements available, the observation of high-risk products indicates that stricter regulations may be warranted to protect consumers from unknowingly ingesting prescription medicine.

1. Introduction

The use of dietary supplements has gained considerable interest over the last three decades. In the US, use is at an all-time high, with 80% of respondents reporting that they took one or more dietary supplements in a 2021 survey (Council for Responsible Nutrition, 2021). Available without a prescription, dietary supplements are an oral addition to the diet, and they are not intended to treat, diagnose, prevent, or cure any disease (Dietary Supplement Health and Education Act of 1994, 1994; New Zealand Government, 1985). While their definitions and regulation can differ greatly between countries, the US currently classifies both nutrients (e.g. vitamins, minerals) and herbal/botanical extracts as dietary supplements, the latter of which are a higher risk to cause adverse effects in users (Dwyer *et al.,* 2018).

As dietary supplements are not approved for therapeutic use, they do not undergo the same level of scrutiny and testing as conventional medicines. In fact, limited pharmacological and toxicological data exist for many herbal products (Petroczi *et al.,* 2011; Ekor, 2014). Despite concerns over product safety and insufficient scientific evidence supporting their use (Starr, 2015), 75% of consumers believe dietary supplements pose no or very little risk while less than half report uses to their physician (Troxler *et al.,* 2013). Consequently, consumer awareness on safe and rational use of dietary supplements is low, with inadequate knowledge on modes of action, potential adverse reactions, contraindications and interactions with food and drug products (Ekor, 2014).

Thanks to a large international market with rapidly changing formulae, current oversight of dietary supplement products remains a challenge. US regulations require producers to follow Current Good Manufacturing Practices (US Food and Drug Administration, 2007). Yet, in 2013, the US Food and Drug Administration (FDA) only inspected 2.8% of registered supplement manufacturers (Long, 2014). Due to such factors, the incidence of hidden ingredients detected in dietary supplements is on the rise (Harel *et al.,* 2013).

Often when products are identified to violate regulations, the FDA issues voluntary recalls (Cohen,

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2018), which have not proven to be entirely effective, as products can remain in the market for years after a recall notice (Cohen *et al.,* 2014). Furthermore, consumers are not always aware of product recalls, even after long term use (Cohen *et al.,* 2012).

Without strict regulation, there is potential for dietary supplements to contain unlabeled contaminants and/or additives (Marcus, 2016; Rocha *et al.,* 2016). Recently, mycotoxins, heavy metals, pesticides, polychlorinated biphenyls, and dioxins have been detected in dietary supplement products (Cole and Fetrow, 2003; Costa *et al.,* 2019). Herbal and botanical supplements remain the highest risk, as 59% of those tested have contained plant species not listed on the label (Newmaster *et al.,* 2013). Additionally, there has been an increase in adulterating herbal supplements with pharmacologically active ingredients (Ekar and Kreft, 2019), as the FDA identified 776 dietary supplements with unapproved pharmaceuticals from 2007 to 2016 (Tucker *et al.,* 2018). The most commonly adulterated products were those marketed towards sexual enhancement and/or weight loss.

Unknowingly consuming pharmaceutical drugs is a serious health risk, since it can lead to adverse side effects or death, especially for at-risk populations. In New Zealand, dietary supplements have been identified to contain both current pharmaceuticals, such as sildenafil (Viagra®) and tadalafil (Cialis®) (Medsafe New Zealand, 2010a), and those banned from the market, such as sibutramine and phenolphthalein (Medsafe New Zealand, 2019). These drugs can induce several health effects, such as manipulation of blood pressure and dizziness, or in the case of sibutramine, an increased risk of cardiovascular events and stroke (Medsafe New Zealand, 2010b).

New Zealand currently follows similar regulations as the US, however, the country has more limited resources for dietary supplement testing and oversight. In some cases, product investigations have uncovered adverse events linked to consumption (Kirkness, 2019). Given the relatively limited product testing in the New Zealand marketplace, this study investigated 68 different dietary supplements marketed in New Zealand specifically at online retail outlets, looking at products advertised for weight loss and sexual enhancement. An LC-MS/MS method was developed and validated for compounds of interest detected in the dietary supplements. With a global need for more comprehensive analytical methods to test herbal dietary supplements (Bailey, 2020), GC-MS and LC-UV methods were also developed and validated for the detection of 9 drug compounds (vardenafil, sildenafil, phenolphthalein, tadalafil, sibutramine, fluoxetine, dapoxetine, yohimbine and caffeine), all of which have been commonly observed over both supplement classes in previous investigations (Medsafe New Zealand, 2010a; Tucker *et al.,* 2018; Medsafe New Zealand, 2019). The results of this study provide key insight into the health and safety risks currently posed to dietary supplement consumers in New Zealand and countries with similar regulatory regimes.

2. Materials and methods

This study investigated the chemical contents of 68 dietary supplements, 32 marketed for weight loss (denoted W1-W32) and 36 marketed for sexual enhancement (denoted E1-E36). All products were advertised specifically in the New Zealand online marketplace during October 2019. Untargeted analysis was performed on each sample using GC-MS and LC-MS techniques while targeted analysis was completed using three validated methods: GC-MS, LC-UV, and LC -MS/MS.

2.1 Equipment

Method development and validation activities focused on three drug compounds that were detected during initial untargeted screening (tadalafil, yohimbine, and caffeine). To further test the specificity of the developed methods and assess its potential for broader use, an additional 6 drug compounds were also included in the method validation activities (Table 1). These compounds were chosen based on their detection in dietary supplements from previous investigations both in New Zealand and globally as well as from untargeted GC-MS and LC-MS screening methods employed in this study (Tucker *et al.,* 2018; Medsafe New Zealand, 2010a; Medsafe New Zealand, 2010b; Medsafe New Zealand, 2010c; Medsafe New Zealand, 2019).

2.2 Liquid chromatography analysis

For LC-UV and LC-MS/MS analyses, mobile phase A was ultra-pure water from a water purification system (Sartorius, Arium®, 18.2 MΩ) containing 0.1% formic acid (Fisher Chemical, Optima Grade) while mobile phase B was acetonitrile (JT-Baker, HPLC Grade). Analyses were performed on an Agilent 1290 LC-UV system coupled to an Agilent 6460 Triple Quad MS detector. A column with a pentafluorophenyl (PFP) F5 stationary phase (Phenomenex, Kinetex®) was used for LC analyses (2.6 µm particle size and column dimensions 50 × 2.1 mm). The column was kept at 40^{o}C and the injection volume was 5 µL. The optimized LC methods had a total flow rate of 0.4 mL/min. The solvent gradient was initially set at 5% acetonitrile, increasing to 25% acetonitrile at 2 mins, then to 40% acetonitrile at 12 mins, followed by an increase to 95% acetonitrile at 17.5

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Table 1. The 9 different compounds that were investigated in the method development and validation.

Name	CAS No.	Trade Name(s)	Classification	Source	Purity (%)
Sildenafil citrate	171599-83-0	Viagra	Erectile Dysfunction (SE)	INADFC	99.74
Tadalafil	171596-29-5	Cialis	Erectile Dysfunction (SE)	INADFC	99.55
Vardenafil $HCl.3H_2O$	224785-90-4	Levitra, Staxyn, Vivanza	Erectile Dysfunction (SE)	INADFC	99.42
Yohimbine HCl	65-19-0	N/A	Erectile Dysfunction (SE)	TRC, Inc.	98
Dapoxetine HCl	119356-77-3	Priligy	Premature ejaculation (SE)	AK Scientific, Inc.	98
Fluoxetine HCl	54910-89-3	Prozac, Sarafem	Premature ejaculation (SE)	AK Scientific, Inc.	95
Sibutramine HCl	106650-56-0	Meridia*	Appetite suppressant (WL)	INADFC	99.36
Phenolphthalein	77-09-8	Ex-Lax*	Laxative (WL)	Ajax Chemical	98
Caffeine	58-08-2	N/A	Stimulant (WL)	Crescent Chemical	99

*These active ingredients have been banned from the New Zealand market. In the case of Ex-Lax, phenolphthalein has been replaced with senna glycoside since 1997 (Coogan *et al.*, 2000).
SE: sexual enhancement, WL: weight loss, INADFC: Indonesia National Agency of Drug and Food Control.

min, where it was held for 2 mins before reverting to initial conditions of 5% acetonitrile at 20 mins and held until 22 mins. Electrospray ionization was used in positive mode with a capillary voltage of 4.0 kV, a gas temperature of 350°C, a gas flow 10 L/min, a sheath gas temperature of 350°C, and a sheath gas flow 12 L/min. The UV detection was applied using a diode array at 4 wavelengths: 254, 270, 280, and 290 nm. For untargeted analysis, the MS was used in scan mode from *m/z* 40 - 800. Further information on the LC-MS/MS detection parameters and transitions are provided in Table 2.

Products that were identified as high risk were sent to the Institute of Environmental and Science Research Limited (ESR) for confirmatory analysis using a Dionex UltiMate™ LC system coupled to a Bruker maXis™ TOF-MS/MS detector. In this confirmatory method, separation was accomplished using a C18 column (Phenomenex Kinetex, 2.6 µm, 100 × 2.1 mm). Mobile phase A was 0.1% formic acid in 98% H_2O/2% acetonitrile. Mobile phase B (98% acetonitrile/2% H_2O, 0.1 % formic acid) was increased from 0% to 80% in 10 min, then to 100% by 12 mins before reverting to 0% at 16 min. Electrospray ionization was used in positive mode with a scan range from *m/z* 50 - 1000.

2.3 Gas chromatography analysis

GC-MS analyses were performed on an Agilent 7890 GC system with a 5795 MSD quadrupole detector using electron impact ionization at 70 eV. The system was equipped with an Agilent DB-5MS column (30 m, 0.25 mm, 0.25 µm). Method development activities yielded an optimized method where the oven temperature was initially held at 70°C for 2 mins before increasing at the rate of 15°C/min to 190°C, followed by a rate of 2.5°C/min to 220°C, and then at a rate of 15°C/min to 280°C, where the temperature was held for 2 mins. Sample injection volume was 1 µL, and the analysis was performed in splitless mode with a helium carrier gas flow of 1 mL/min. The injector, MS transfer line, MS source and MS quad were set at 250°C, 280°C, 230°C, and 150°C, respectively. The MS was scanned from m/z 40 - 800. While the GC and LC methods were developed for this study, the methods detailed in Rindelaub *et al.* (2019) were used as a starting point for the method development activities.

2.4 Extraction procedure

Supplements were extracted in 7:3 methanol:acetonitrile after it was determined that sildenafil and tadalafil were only slightly soluble in pure methanol (Macron, ChromAR®). For analysis, 50±5 mg of each sample was weighed into a microcentrifuge tube and 1.0 mL of the extraction solvent was added. The sample was mixed using a vortex mixer for 30 s and then placed in an ultrasonic bath for 30 s. The contents were centrifuged at 4000 rpm for 3 mins to separate the sample. If separation could not be accomplished, the supernatant was filtered through a syringe filter (PTFE, 0.22 µm). Extraction methods were developed from methods presented in Cho *et al.* (2014) and Zheng *et al.* (2016).

For 10 randomly selected encapsulated samples, the shell of the capsule was also tested separately for adulterants using GC-MS. Sample capsules were broken and the filling was poured out to leave only the empty capsule shell. Each shell was homogenized using a mortar and pestle prior to analysis. The emptied capsules were dissolved in 20 mL of phosphate buffer at pH 6.4 and then a liquid-liquid extraction was performed with dichloromethane (JT Baker, analytical grade). The

Table 2. The MRM transitions (*m/z*) and collision induced dissociation (CID) energy used for the LC-MS/MS method.

Analyte	Precursor ion (*m/z*)	Quantitative ion (*m/z*)	Qualifier ion (*m/z*)	CID energy (eV)
Tadalafil	390	268	135	20
Yohimbine	355	144	212	25
Caffeine	195	138	110	20

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phosphate buffer solution was added to a 100 mL separatory funnel and extracted with 10 mL of dichloromethane in triplicate. The dichloromethane extraction solutions were combined and then injected directly onto the GC-MS using the method described in Section 2.3. The shell capsule extraction method was developed based on Venhuis *et al.* (2012).

2.5 Untargeted analysis

In addition to targeted analysis of the analytes in Table 1, this study also used full scan MS mode in each the GC and LC experiments to screen for possible adulterants, analogues, and impurities. In GC-MS analyses, the resulting mass spectra were compared to the NIST spectral database or an authentic standard where available (e.g. caffeine, yohimbine) while in LC-MS analyses the parent ion in positive mode (e.g. [M+H]) was compared to the known masses of common dietary supplement ingredients when standards were unavailable. Analytical methods were then developed and validated to include the analytes of interest discovered in the untargeted analysis along with the drugs listed in Table 1 to ensure that the developed methods could be used in the analysis of a wide range of possible adulterant compounds. The final validated methods are described in Sections 2.2 and 2.3.

2.4 Validation parameters

The optimized LC-MS/MS, LC-UV, and GC-MS methods were evaluated for specificity, linearity, accuracy, precision, intermediate precision, and quantification/detection limits (LOQ/LOD), as per guidance from the International Conference on Harmonisation (ICH Harmonised Tripartite Guideline, 2005; Kruve *et al.,* 2015a). During validation, standards were spiked into a matrix that contained a combination of homogenized dietary supplement samples, such as those containing herbal extracts from *Epimidium*, *Butea superba*, *Panax ginseng*, *Garcinia cambogia*, Zingiber (sexual enhancement), and açaí berry (weight loss). These matrices were selected after an initial screening experiment did not reveal the presence of any of the targeted compounds.

2.4.1 Specificity

Specificity was defined as the ability to unambiguously discriminate the compound of interest from the other analytes and any method or matrix impurities. Due to their molecular functionality, only caffeine, sibutramine, fluoxetine, and dapoxetine could be detected using the GC-MS technique. In each of the validations, spiked samples and a matrix blank sample were extracted and injected separately on the instruments. Extracted ion current chromatograms (EICs) for each species were used for identification and quantification in the GC-MS validation activities while absorption at select wavelengths was used for the LC-UV validation (Table 1). To meet specificity requirements in this study, signals from the other analytes of interest or those from the matrix blank should not contribute to more than 20% of the limit of quantification of the given analyte (Kruve *et al.,* 2015b).

2.4.2 Linearity

For linearity, six working standards were created for each analyte of interest and injected in duplicate on the respective instrument. The resulting calibration curve linear regression required a correlation coefficient (R) ≥ 0.99. The concentrations of the working standards ranged between 0.20 - 50 µg/mL for GC-MS analysis, 0.50 - 8.0 µg/mL for LC-UV analysis, and 0.0092 - 0.62 µg/mL for LC-MS/MS analysis.

2.4.3 Accuracy, precision, and intermediate precision

Evaluation of accuracy and precision were done in parallel by spiking matrix samples with each analyte at 3 different concentrations and then injecting the subsequent extracts in triplicate on the instrument. For accuracy in this study, the recovered amount of each injection was required to be within 80 - 120% of the theoretical concentration while precision required the relative standard deviation (RSD) of the determined peak areas from the triplicate injections to be ≤15% and the retention times to be ≤2%. Intermediate precision, which had the same evaluation criteria as precision, was determined by repeating the precision process on a different day. Quantification of analytes in each dietary supplement sample was accomplished using the LC-UV method (described in Section 2.2), due to the superior precision, accuracy, and linearity determined for the validated UV detection method.

2.4.4 Limit of quantification and limit of detection

The limit of quantification (LOQ) and the limit of detection (LOD) were defined by the following equations:

$$LOQ = 10\ \sigma / S \quad (1)$$

$$LOD = 3.3\ \sigma / S \quad (2)$$

Where S is the slope of the calibration curve determined and σ is the standard deviation of the peak area from triplicate injections of the lowest concentration standard for each analyte.

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3. Results

3.1 Method validation

The LC-MS/MS method was validated for tadalafil, yohimbine, and caffeine (Figure 1), the main compounds of interest in this study. While outside the focus of this study, methods for the analysis of six other drug compounds (vardenafil, sildenafil, phenolphthalein, sibutramine, fluoxetine, and dapoxetine) were validated across GC-MS and LC-UV techniques, alongside the compounds of interest. Further information on the GC-MS and LC-UV validations are provided in Tables S2 and S3, respectively.

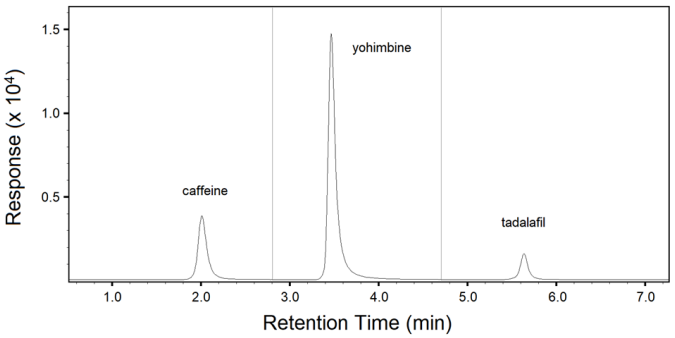

Figure 1. An LC-MS/MS chromatogram of caffeine, yohimbine, and tadalafil.

All analytes of interest were separated efficiently using LC-MS/MS, as shown in Figure 1, and the resulting validation parameters were all within the specific limits (Table 3). A method comparison for the analytes of interest is provided in Table 4, with the best precision, accuracy, and linearity observed for the LC-UV method.

3.2 Dietary supplements

3.2.1 Untargeted analysis

The untargeted analysis of analytes was completed using both the GC-MS and LC-MS scanning methods. While the GC-MS results mostly yielded ester species related to fatty acids, several unlabeled compounds were tentatively identified using the GC-MS and LC-MS methods (Tables 4 and 5). The most toxicologically concerning of these were tadalafil and yohimbine. Tadalafil was identified in one sexual enhancement product (E24) while yohimbine was identified in 3 sexual enhancement products (E8, E11, E25). Caffeine was also identified in 13 products (11 marketed for weight loss and two marketed for sexual enhancement). Further information on each drug detected is provided below.

The emptied supplement capsules of ten dietary supplements (W7, W8, W13, W14, W18, E1, E5, E25, E26 and E27) were tested separately, and none yielded chromatographic peaks corresponding to targeted analytes of interest (tadalafil, yohimbine, caffeine, sildenafil, vardenafil, phenolphthalein, sibutramine, fluoxetine and dapoxetine).

Table 4. A comparison of the validation parameters determined for caffeine using each method (GC-MS, LC-UV, LC-MS/MS).

Analyte	GC-MS	LC-UV	LC-MS/MS
Precision (% RSD)	8.7	0.28	3.6
Accuracy (% Recovery)	105	94	119
Linearity (R value)	0.9922	0.9996	0.9937

3.2.2 Tadalafil

The pharmaceutical drug tadalafil (Cialis®) was found as an unlisted ingredient in one (E24) of the 36 sexual enhancement dietary supplements tested. The dietary supplement claimed to be manufactured in New Zealand, and its capsule contained a white powder, which was in contrast to other herbal extracts that were either brown, yellow or green in color. Side effects of tadalafil use range from muscle pain and nausea to serious issues like priapism and vision loss. Additionally, tadalafil should not be used in conjunction with nitrate heart medication as it could induce a fatally low drop in blood pressure. Commercially, tadalafil doses range from 5-20 mg per day while, in this study, one capsule of the adulterated product contained 25.2±1.1 mg tadalafil. The amount of tadalafil observed is comparable to its maximum daily recommended intake (20 mg) (Medsafe New Zealand, 2022). Since the packaging stated that no more than two capsules should be consumed in a 24 hrs period, it is possible that an individual could consume more than double the maximum daily recommended dose of tadalafil using this product. The potential for serious side effects and the amount of the adulterant observed in this over-the-counter product indicate that this dietary supplement is high risk.

Table 3. The validation parameters for the LC-MS/MS validation of caffeine, yohimbine, and tadalafil

	Accuracy 80-120%		Precision ≤2%	Precision ≤15%	Intermediate Precision ≤2%	Intermediate Precision ≤15%	Linearity R > 0.99		Limits ng/mL	
	RT	% Recovery	RT RSD %	Area RSD %	RT RSD %	Area RSD %	Slope × 10^{-5}	R value	LOQ	LOD
Caffeine	2.00	119	0.17	3.61	0.16	1.86	2.4	0.994	17	5.8
Yohimbine	3.47	109	0.03	1.24	0.06	2.88	52.2	0.994	4.9	1.6
Tadalafil	5.65	109	0.03	2.71	0.14	1.77	1.49	0.994	6.8	2.3

RT: retention time (min), RSD: relative standard deviation.

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Table 5. Details on the caffeine detected in dietary supplements.

Sample	Caffeine content in each capsule/tablet/ bag (mg)	Label information on caffeine content	Max. daily use (as per label)
E18	4.5±0.3	Not mentioned	6 capsules
E19	4.0±0.3	Mentions green tea extract	5 capsules
W20	7.6±0.4	1 capsule contains 8.5 mg	2 capsules
W21	70.8±6.8	1 serving contains 200 mg	2 servings (6 capsules)
W22	50.1±2.0	3 capsules contain 200 mg	6 capsules
W26	1.4±0.2	Mentions green tea extract	2 capsules
W3	249.6±9.0	1 capsule contains 201 mg	1 capsule
W5	1.6±0.2	Mentions green tea extract	2 capsules
W6	106.9±10.9	2 tablets contain 186.6 mg	2 servings (4 tablets)
W8	18.0±0.8	4 capsules contain 75 mg	2 servings (8 capsules)
W30	166.5±7.5	2 capsules contain 390 mg	2 capsules
W31	3.2±0.7	Mentions caffeine from green tea	2 tea bags
W32	85.6±5.1	1 capsule contains 70 mg	3 capsules

3.2.3 Yohimbine

Yohimbine was detected in 3 of the 26 sexual enhancement supplements tested. Yohimbine, derived from the African tree *Pausinystalia johimbe*, is a medicine in New Zealand that is only available by prescription. Due to potential adverse side effects, such as heart, kidney, and liver problems, it has been banned for consumption in the UK, Australia, The Netherlands, and Canada (Panel on Food Additives and Nutrient Sources added to Food, 2013). One capsule of each E8, E11, and E25 contained 1.3, 9.9, and 2.0 mg of yohimbine, respectively. In the case of sample E11, the label recommended dosage would correspond to consumption of 19.8 mg yohimbine, which is greater than the 16.2 mg daily dose given to patients in the first stage of a previously published clinical trial (Guay *et al.,* 2002).

3.2.4 Caffeine

While no pharmaceutical adulterants were detected in the weight loss supplements, the analysis revealed that 11 of the 32 samples contained caffeine, in addition to two of the 33 sexual enhancement supplements (Table 5). The largest amount of caffeine found per item was 250±9 mg in sample W2, whose label claimed to have 201 mg per capsule. The maximum recommended daily intake of caffeine is 400 mg (EFSA Panel on Dietetic Products, Nutrition and Allergies (NDA), 2015), however, the maximum daily use of products W6 and W21 as per their labeled guidelines would result in consumption of 428±44 mg and 425±41 mg caffeine, respectively.

Of the products that listed caffeine as an ingredient, the determined amounts were within 25% of the label claim. Low levels of caffeine were found in the four products that had green tea extract on the label information (<5 mg per capsule). Caffeine was also found in one sexual enhancement supplement where neither caffeine nor a tea extract were listed on the product label. The maximum intake of caffeine from this product, based on label guidelines, was 27±2 mg. Although loosely regulated, the use of caffeine from energy, pre-workout, and weight loss products is linked to a significant increase in serious adverse effects, including death, compared to the use of noncaffeinated products (Jagim *et al.,* 2020).

4. Discussion

The results of this study add to the growing data set on unlabeled additives found in dietary supplements across the global marketplace (Tucker *et al.*, 2018). Synthetic compounds and illicit herbal materials can be added to dietary supplements to increase their desired effects (Biesterbos *et al.*, 2019), however, such actions could put unassuming consumers at risk. For instance, the consumption of the prescription medications detected in this study (tadalafil and yohimbine) could lead to organ damage or even death in vulnerable populations (Nagasawa *et al.*, 2021). While this study cannot determine if such adulterants were intentionally added to the products, a synthetic prescription medication (e.g. tadalafil) would not be a likely contaminant in a facility that exclusively produces herbal supplements. Further investigation into the production of this dietary supplement is warranted.

Since this study only focused on products advertised and sold online, it should not be considered a complete representation of the New Zealand market. Further investigations into the products available at health/ nutrition retailers, pharmacies, supermarkets, gyms, and adult entertainment stores would be helpful to understand how the digital market compares to the physical market, where products can be shielded from

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online scrutiny. This study shows that further investigation of dietary supplements is needed, especially sexual enhancement products, where 11% of the dietary supplements tested contained unapproved ingredients. Other supplement categories that may warrant testing include muscle building, antidiabetic, pain and fatigue treatment, blood pressure, and cholesterol support.

5. Conclusion

Analysis of dietary supplements marketed online in New Zealand revealed the presence of unlisted drug ingredients in select products. 11% of the sexual enhancement supplements tested contained a substance only available by medical prescription in New Zealand, including tadalafil (Cialis®) and yohimbine. While none of the weight loss products tested contained unapproved substances, large amounts of caffeine were detected in several samples. Two weight loss products had labeled dosages of caffeine that corresponded to levels above the maximum daily recommendations while one sexual enhancement product contained caffeine despite the substance not being listed on the product label. As this study only represents a subset of dietary supplements available in New Zealand, further testing of the marketplace is urgently needed to determine the extent that unlabeled ingredients are present in dietary supplements across New Zealand.

Conflict of interest

The authors declare no conflicts of interest.

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