

United States Department of Agriculture  
Agricultural Marketing Service | National Organic Program  
Document Cover Sheet

<https://www.ams.usda.gov/rules-regulations/organic/petitioned-substances>

Document Type:

**National List Petition or Petition Update**

A petition is a request to amend the USDA National Organic Program's National List of Allowed and Prohibited Substances (National List).

Any person may submit a petition to have a substance evaluated by the National Organic Standards Board (7 CFR 205.607(a)).

Guidelines for submitting a petition are available in the NOP Handbook as NOP 3011, National List Petition Guidelines.

Petitions are posted for the public on the NOP website for Petitioned Substances.

**Technical Report**

A technical report is developed in response to a petition to amend the National List. Reports are also developed to assist in the review of substances that are already on the National List.

Technical reports are completed by third-party contractors and are available to the public on the NOP website for Petitioned Substances.

Contractor names and dates completed are available in the report.

## National List Petition

### Use of magnesium carbonate E504(i) as a processing aid in the manufacture of the soluble extract of organic chicory

**Petitioner :** LEROUX SA  
84 rue François Herbo  
59310 ORCHIES  
FRANCE

**Context :**

Leroux company is specialized in the transformation of chicory root (*Cichorium intybus L.*) in the form of instant product (instant drinks).

Chicory roots washed, sliced, dried, roasted, milled, sieved, and extracted with hot water. Instant chicory powder is obtained by dehydration (spray drying) of this chicory liquid extract.

Leroux has been processing chicory root for over 160 years. <https://www.leroux.com/en>

However, during our last processing steps: atomization and packaging, the instant chicory powder tends to stick to the walls of the installations. This leads to many stops for cleaning and therefore a reduction in production rates. These phenomena are explained by poor fluidity of the powder and therefore a low flow index. In order to overcome this problem, Leroux uses magnesium carbonate, authorized in conventional agriculture. We would like this technological aid (magnesium carbonate) to be included in the list of technological aids authorized in organic products.

Item A.1 — Indicate which section or sections the petitioned substance will be included on and/or removed from the National List. The current National List may be viewed at [www.ams.usda.gov/NOPNationalList](http://www.ams.usda.gov/NOPNationalList).

Name of additive / substance	Primary use/conditions
E504(i): Magnesium Carbonate	Drying agent / Anti-caking agent
§ 205.605: Nonagricultural (nonorganic) substances allowed in or on processed products labelled as “organic” or “made with organic (specified ingredients)”.	

Item A.2

OFPA Category	Processing aids
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Item A.3

Inert Ingredients	Non applicable
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Item B—Information on the substance being petitioned

Item B.1. Substance Name

<i>Chemical name(s)</i>	(i) Magnesium carbonate
<i>Other names</i>	(i) NA
<i>Trade name</i>	Magnesium carbonate
<i>Synonyms</i>	INS No. 504(i)
<i>Definition</i>	A basic hydrated or a normal hydrated magnesium carbonate or a mixture of the two
<i>C.A.S. number</i>	546-93-0
<i>Assay</i>	Not less than 24.0% and not more than 26.4% of Mg
<i>Description</i>	Odourless, light, white friable masses or as a bulky white powder
<i>Functional uses</i>	Anticaking agent

Item B.2. Petitioner and Manufacturer Information

<i>Petitioner</i>	LEROUX 84 rue François Herbo 59310 ORCHIES (France) Phone : 0033 3 20 64 48 00
<i>Manufacturer of magnesium carbonate</i>	Dr. Paul Lohmann France SARL Boulevard de l'Yerres 52 91000 Évry (FRANCE) Phone : 0033 1 81 07 24 40

Item B.3. Intended or Current Use

	Use as a processing aid in the manufacture of organic instant chicory
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**Item B.4. Intended Activities and Application Rate**

Processing aid used during transformation process: magnesium carbonate E504(i).

Magnesium is an alkali metal, it is the eighth most common element in the Earth's crust. Pure magnesium reacts strongly in air or in combination with other elements and is therefore mainly available in nature in ionized form as carbonate for example. In addition to the possibility of enrichment, the technological functions of magnesium salts also bring their benefits. Used as anti-caking agents to improve fluidity, magnesium salts are generally accepted as additives without quantitative restrictions for many food products (*quantum satis*). Magnesium carbonate E504(i) is also authorised by the EU as a processing aid as an anti-caking agent.

<b>Producer/Supplier</b>	Dr Paul Lohmann
<b>Article</b>	Magnesium carbonate
<b>CAS No.</b>	546-93-0
<b>Chemical composition</b>	MgCO <sub>3</sub>
<b>Molecular weight</b>	84.3 g/mol
<b>Appearance</b>	white powder without odor
<b>Mode of action</b>	anticaking agent

**Characteristics**

Parameters	Value
Packed density	120 – 250 g/l
pH (1% suspension)	approx. 10
Solubility (20°C)	<1g in 100ml of water
Magnesium content	Approx. 25%
Humidity (2 hours at 105°C)	Max 2%

**Modalities and doses of use** The use of magnesium carbonate as a processing aid is intended for the manufacture of instant chicory extract obtained by atomization. The incorporation of the E504(i) is done in the air ring at the bottom of the tower feeding the dryer in order to obtain a re-aeration and a very good homogeneity of the product. **The maximum quantity used would be 0.05%.**

**Basic topological data (ADI level)** *ADI level*; Not applicable  
*E504 (i)*: An ADI 'not limited' was established at the 9th JECFA (1965)

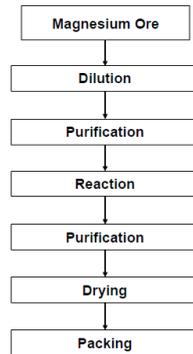
**Date of JECFA /SCF evaluation** E504 (i): Prepared at the 17th JECFA (1973), published in FNP 4 (1978) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004).

**Short summary** Magnesium is an alkali metal

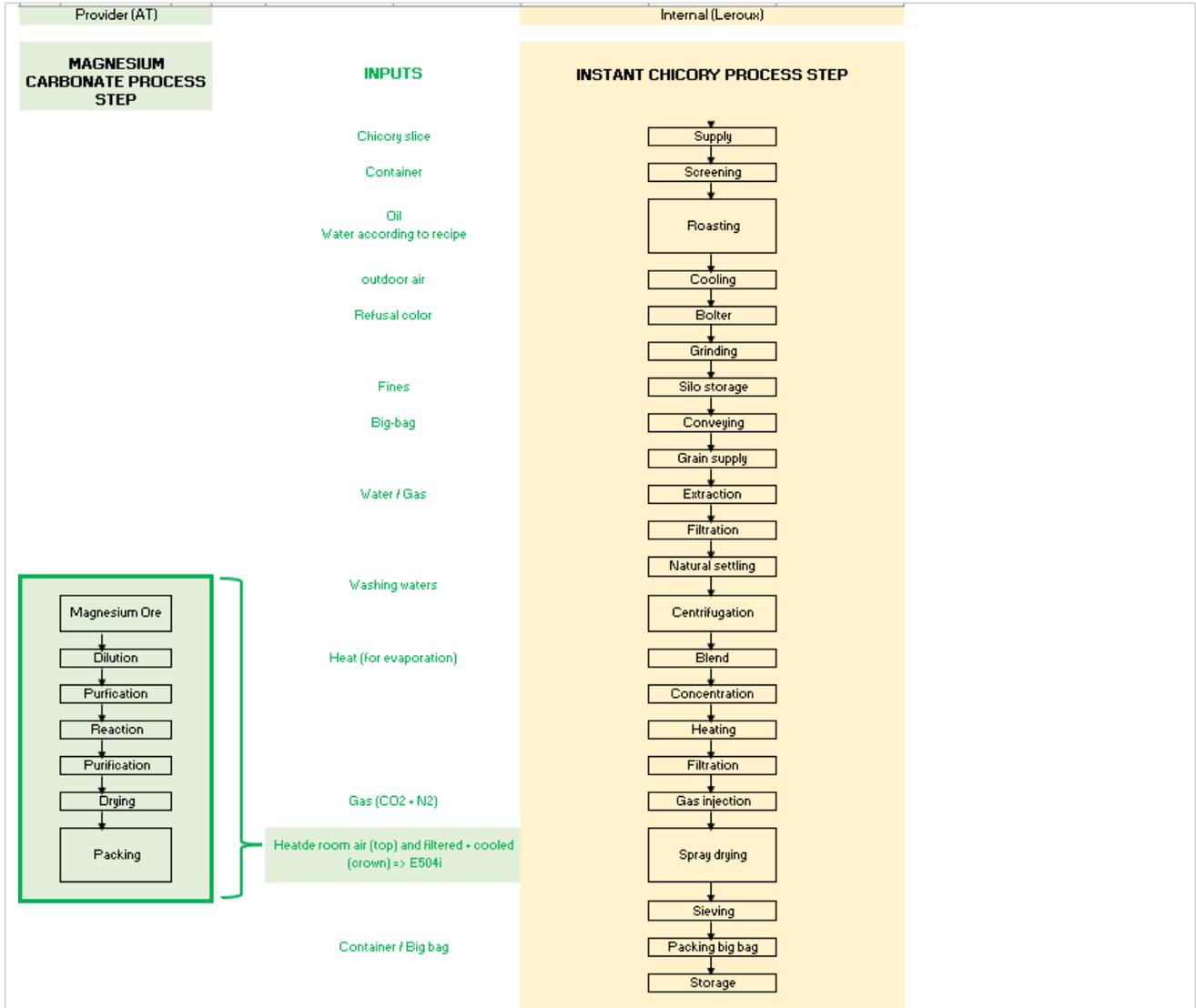
**Item B.5. Manufacturing Process**

<b>Active ingredients:</b>	Magnesium carbonate
<b>Possible carriers</b>	Not applicable
<b>Origin of raw materials (including aspects of mining/harvesting them), production methods</b>	Magnesium is an alkali metal, it is the eighth most common element in the earth's crust. Pure magnesium reacts strongly in air or in association with other elements and is therefore mainly available in nature in ionized form such as carbonate for example. E504 (i) is of inorganic nature.
<b>Source of the substance</b>	Mineral

The diagram of the production of magnesium carbonates is as follows:

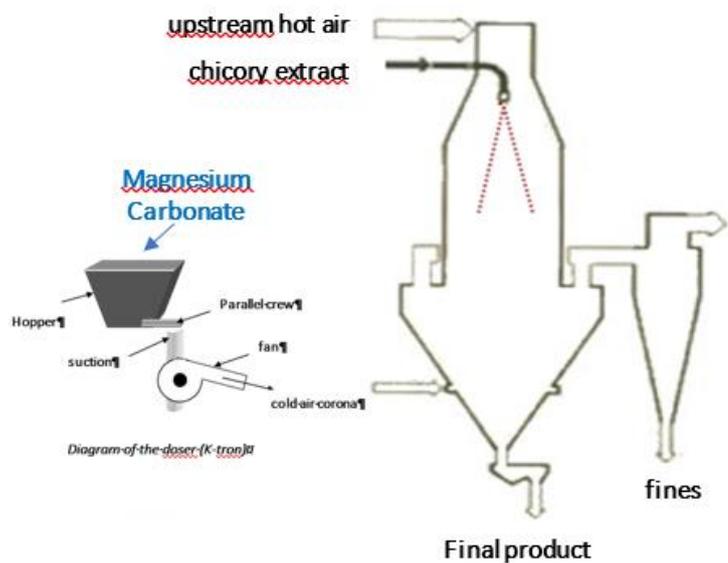


<b><i>Application during food processing</i></b>	E504(i) is used during processing of liquid chicory into soluble powder
<b><i>Intended use</i></b>	The use of E504(i) as a processing aid in the production of soluble chicory extract improves the fluidity and therefore the flow of the powder
<b><i>Technological function in food</i></b>	E504(i) is used as a processing aid : anti-caking agent
<b><i>Technology</i></b>	The use of magnesium carbonate as a processing aid is intended for the manufacture of the instant extract of chicory obtained by atomization. The incorporation of E504(i) is done in the crown of air at the bottom of the tower feeding the dryer in order to obtain re-aeration and very good homogeneity of the product (figures 1 and 2). The maximum amount used would be 0.05%.
<b><i>Instant chicory flow chart</i></b>	



Instant chicory process

Schematic of a Tall form (Niro) atomizing tower



**Item B.6. Ancillary Substances**

	Not applicable
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**Item B.7. Previous Reviews**

	<p>[Code of Federal Regulations] [Title 21, Volume 6] [CITE: 21CFR582.1425]</p>
<b><i>NOSB Review Documents</i></b>	<ul style="list-style-type: none"> <li>• 1996 NOSB review for addition to §205.605 for use as a processing aid</li> <li>• 1996 <a href="#">Technical Advisory Panel (pdf)</a></li> <li>• September 1996 <a href="#">NOSB Recommendation (pdf)</a> (page 392) (synthetic, nonsynthetic)</li> <li>• November 2005 <a href="#">Petition (pdf)</a> requesting addition to §205.605 for use as a processing aid (filtering aid)</li> <li>• October 2015 <a href="#">NOSB Subcommittee Proposal (pdf)</a> (page 20)</li> <li>• October 2015 <a href="#">NOSB Recommendation (pdf)</a> (page 27)</li> </ul>

**Item B.8. Regulatory Authority**

	<p>[Code of Federal Regulations] [Title 21, Volume 6] [CITE: 21CFR582.1425]</p> <p>TITLE 21--FOOD AND DRUGS CHAPTER I--FOOD AND DRUG ADMINISTRATION DEPARTMENT OF HEALTH AND HUMAN SERVICES SUBCHAPTER E - ANIMAL DRUGS, FEEDS, AND RELATED PRODUCTS PART 582 -- SUBSTANCES GENERALLY RECOGNIZED AS SAFE Subpart B - General Purpose Food Additives Sec. 582.1425 Magnesium carbonate.</p> <p>(a) Product. <b>magnesium carbonate</b> .</p> <p>(b) Conditions of use. This substance is generally recognized as safe when used in accordance with good manufacturing or feeding practice.</p>
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<p><a href="https://echa.europa.eu/fr/registration-dossier/-/registered-dossier/15234/5/5/1">https://echa.europa.eu/fr/registration-dossier/-/registered-dossier/15234/5/5/1</a></p>	<p>the water and is necessary to maintain a good chemical balance in soils, water and plants, and carbonate will become part of the carbon cycle.</p> <p><b>Hydrolysis</b> : No studies are available. A study is not required, because magnesium carbonate is a simple inorganic salt.</p> <p><b>Biodegradation</b> : Magnesium carbonate is an inorganic substance and will not undergo biodegradation.</p> <p><b>Bioaccumulation</b> : Magnesium carbonate is an inorganic ionic solid for which an octanol/water partition coefficient cannot be reliably determined. Magnesium carbonate dissociates into the magnesium Mg<sup>2+</sup> and carbonate CO<sub>3</sub><sup>2-</sup> ions at environmental pH. These are essential to all living organisms (flora and fauna) and their intracellular and extra-cellular concentrations are actively regulated. Bioaccumulation is therefore not expected.</p> <p><b>Transport and distribution</b> : Soil adsorption/ desorption studies with magnesium carbonate are not possible due to analytical difficulties, the solubility characteristics of magnesium carbonate in water and its inorganic nature. The distribution coefficient (Kd) for magnesium is 234 -1820.</p>																					
<p><b>Environmental impacts during processing</b></p>	<p>The use of E504(i) as a processing aid in the production of soluble chicory extract improves the fluidity and therefore the flow of the powder. Favored flow generates a reduction in fouling in production and packaging facilities and consequently production compatible with industrial expectations (reduction of material losses and rationalization of cleaning and the associated quantity of water). The environmental impact can be approximated on the basis of the data shown in the table below:</p> <p>Comparative production data with or without processing aid :</p>																					
	<table border="1"> <thead> <tr> <th></th> <th>Without processing aid</th> <th>With processing aid</th> </tr> </thead> <tbody> <tr> <td><b>Installation flow</b></td> <td>max. Base x0.8</td> <td>= Base</td> </tr> <tr> <td></td> <td colspan="2">The impact on energy consumption cannot be accurately projected to date. However, it can be considered that stopping the installations for cleaning increases energy consumption (a cleaning cycle requires a cooling cycle and then reheating the equipment).</td> </tr> <tr> <td><b>Material loss</b></td> <td>Base x8</td> <td>= Base</td> </tr> <tr> <td></td> <td colspan="2">The material loss differential induces an overconsumption of agricultural material in the order of 15 to 20%</td> </tr> <tr> <td><b>Volumes of water consumed for cleaning installations</b></td> <td>Base x3</td> <td>= Base</td> </tr> <tr> <td></td> <td colspan="2">The overconsumption of water could represent up to more than 5000m<sup>3</sup> in one year.</td> </tr> </tbody> </table>		Without processing aid	With processing aid	<b>Installation flow</b>	max. Base x0.8	= Base		The impact on energy consumption cannot be accurately projected to date. However, it can be considered that stopping the installations for cleaning increases energy consumption (a cleaning cycle requires a cooling cycle and then reheating the equipment).		<b>Material loss</b>	Base x8	= Base		The material loss differential induces an overconsumption of agricultural material in the order of 15 to 20%		<b>Volumes of water consumed for cleaning installations</b>	Base x3	= Base		The overconsumption of water could represent up to more than 5000m <sup>3</sup> in one year.	
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<p><b>Effects on Human health</b></p>	<p>Not applicable</p>																					
<p><b>Effects on soil organisms, crops, or livestock</b></p>	<p>Not applicable</p>																					
<p><b>Food quality and authenticity r</b></p>	<p>Not applicable</p>																					

Item B.11. Safety Information

<p> Dr. Paul Lohmann® Printing date 03.02.2020</p>	<p><i>Safety data sheet</i> according to 1907/2006/EC, Article 31 Version number 5</p>	<p>Page 1/6 Revision: 26.11.2019</p>
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**SECTION 1: Identification of the substance/mixture and of the company/undertaking**

- **1.1 Product identifier**
- **Trade name:** Magnesium carbonate
- **Article number:** C36 E504(i)
- **CAS Number:**  
546-93-0
- **EC number:**  
208-915-9
- **Registration number**  
*For traceability reasons, the REACH registration number is available on request. If you have a use that requires a registration according to REACH, please contact our sales department.*
- **1.2 Relevant identified uses of the substance or mixture and uses advised against**  
*No further relevant information available.*
- **Application of the substance / the mixture**  
Food additive  
Pharma Active ingredients
- **1.3 Details of the supplier of the safety data sheet**
- **Manufacturer/Supplier:**  
Dr. Paul Lohmann GmbH & Co. KGaA  
Hauptstraße 2  
D-31860 Emmerthal  
GERMANY  
MSDS@lohmann-chemikalien.de
- **Further information obtainable from:** Product safety department
- **1.4 Emergency telephone number:**  
Emergency CONTACT (24-Hour-Number): GBK GmbH +49 (0)6132-84463

**SECTION 2: Hazards identification**

- **2.1 Classification of the substance or mixture**
- **Classification according to Regulation (EC) No 1272/2008**  
*The substance is not classified, according to the CLP regulation.*

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- **2.2 Label elements**
- **Labelling according to Regulation (EC) No 1272/2008** Void
- **Hazard pictograms** Void
- **Signal word** Void
- **Hazard statements** Void
- **2.3 Other hazards**
- **Results of PBT and vPvB assessment**
- **PBT:** Not applicable.
- **vPvB:** Not applicable.

**SECTION 3: Composition/information on ingredients**

- **3.1 Chemical characterisation: Substances**
- **CAS No. Description**  
546-93-0 Magnesium carbonate  
Alternative CAS number: 23389-33-5
- **Identification number(s)**
- **EC number:** 208-915-9

**SECTION 4: First aid measures**

- **4.1 Description of first aid measures**
- **General information:** No special measures required.
- **After inhalation:** Supply fresh air; consult doctor in case of complaints.

(Contd. on page 2)

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**Trade name: Magnesium carbonate**

(Contd. of page 1)

- **After skin contact:** Generally the product does not irritate the skin.
- **After eye contact:**  
Rinse opened eye for several minutes under running water. If symptoms persist, consult a doctor.
- **After swallowing:** If symptoms persist consult doctor.
- **4.2 Most important symptoms and effects, both acute and delayed** No further relevant information available.
- **4.3 Indication of any immediate medical attention and special treatment needed**  
No further relevant information available.

### **SECTION 5: Firefighting measures**

- **5.1 Extinguishing media**
- **Suitable extinguishing agents:** Use fire extinguishing methods suitable to surrounding conditions.
- **5.2 Special hazards arising from the substance or mixture** No further relevant information available.
- **5.3 Advice for firefighters**
- **Protective equipment:**  
Do not inhale explosion gases or combustion gases.  
Wear self-contained respiratory protective device.

### **SECTION 6: Accidental release measures**

- **6.1 Personal precautions, protective equipment and emergency procedures** Wear protective clothing.
- **6.2 Environmental precautions:** Do not allow to enter sewers/ surface or ground water.
- **6.3 Methods and material for containment and cleaning up:**  
Pick up mechanically.  
Send for recovery or disposal in suitable receptacles.
- **6.4 Reference to other sections**  
See Section 7 for information on safe handling.  
See Section 8 for information on personal protection equipment.  
See Section 13 for disposal information.

### **SECTION 7: Handling and storage**

- **7.1 Precautions for safe handling** No special precautions are necessary if used correctly.
- **Information about fire - and explosion protection:** The product is not flammable.
- **7.2 Conditions for safe storage, including any incompatibilities**
- **Storage:**
- **Requirements to be met by storerooms and receptacles:** No special requirements.
- **Information about storage in one common storage facility:** Not required.
- **Further information about storage conditions:** Store in cool, dry conditions in well sealed receptacles.
- **7.3 Specific end use(s)** No further relevant information available.

### **SECTION 8: Exposure controls/personal protection**

- **Additional information about design of technical facilities:** No further data; see item 7.
- **8.1 Control parameters**
- **Ingredients with limit values that require monitoring at the workplace:** Not required.
- **Additional information:** The lists valid during the making were used as basis.
- **8.2 Exposure controls**
- **Personal protective equipment:**
- **General protective and hygienic measures:**  
The usual precautionary measures are to be adhered to when handling chemicals.  
Keep away from foodstuffs, beverages and feed.  
Do not eat, drink, smoke or sniff while working.

(Contd. on page 3)

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**Trade name: Magnesium carbonate**

(Contd. of page 2)

- *Be sure to clean skin thoroughly after work and before breaks.*
- **Respiratory protection:** Use suitable respiratory protective device in case of insufficient ventilation.
- **Protection of hands:**



*Protective gloves*

*The glove material has to be impermeable and resistant to the product/ the substance/ the preparation. Due to missing tests no recommendation to the glove material can be given for the product/ the preparation/ the chemical mixture.*

*Selection of the glove material on consideration of the penetration times, rates of diffusion and the degradation*

**- Material of gloves**

*The selection of the suitable gloves does not only depend on the material, but also on further marks of quality and varies from manufacturer to manufacturer.*

**- Penetration time of glove material**

*The exact break through time has to be found out by the manufacturer of the protective gloves and has to be observed.*

- **Eye protection:** Safety glasses
- **Body protection:** Protective work clothing

**SECTION 9: Physical and chemical properties**

**- 9.1 Information on basic physical and chemical properties**

**- General Information**

**- Appearance:**

<b>Form:</b>	<i>Powder</i>
<b>Colour:</b>	<i>White</i>
<b>Odour:</b>	<i>Nearly odourless</i>

**- pH-value (50 g/l) at 20 °C:** *10-11*

**- Change in condition**

<b>Melting point/freezing point:</b>	<i>Undetermined.</i>
<b>Initial boiling point and boiling range:</b>	<i>Undetermined.</i>

**- Flash point:** *Not applicable.*

**- Flammability (solid, gas):** *Product is not flammable.*

**- Explosive properties:** *Product does not present an explosion hazard.*

**- Density:** *Not determined.*

**- Solubility in / Miscibility with water:** *Insoluble.*

**- 9.2 Other information** *No further relevant information available.*

**SECTION 10: Stability and reactivity**

**- 10.1 Reactivity** *No further relevant information available.*

**- 10.2 Chemical stability**

**- Thermal decomposition / conditions to be avoided:**

*To avoid thermal decomposition do not overheat.*

*No decomposition if used and stored according to specifications.*

**- 10.3 Possibility of hazardous reactions** *Reacts with acids.*

**- 10.4 Conditions to avoid** *No further relevant information available.*

**- 10.5 Incompatible materials:** *No further relevant information available.*

(Contd. on page 4)

*Trade name: Magnesium carbonate*

(Contd. of page 3)

- **10.6 Hazardous decomposition products:** *No dangerous decomposition products known.*

### **SECTION 11: Toxicological information**

- **11.1 Information on toxicological effects**
- **Acute toxicity** *Based on available data, the classification criteria are not met.*
- **Primary irritant effect:**
- **Skin corrosion/irritation** *Based on available data, the classification criteria are not met.*
- **Serious eye damage/irritation** *Based on available data, the classification criteria are not met.*
- **Respiratory or skin sensitisation** *Based on available data, the classification criteria are not met.*
- **Toxicokinetics, metabolism and distribution** *Undetermined.*
- **Acute effects (acute toxicity, irritation and corrosivity)** *Undetermined.*
- **Sensitisation** *Undetermined.*
- **Repeated dose toxicity** *Undetermined.*
- **CMR effects (carcinogenicity, mutagenicity and toxicity for reproduction)**
- **Germ cell mutagenicity** *Based on available data, the classification criteria are not met.*
- **Carcinogenicity** *Based on available data, the classification criteria are not met.*
- **Reproductive toxicity** *Based on available data, the classification criteria are not met.*
- **STOT-single exposure** *Based on available data, the classification criteria are not met.*
- **STOT-repeated exposure** *Based on available data, the classification criteria are not met.*
- **Aspiration hazard** *Based on available data, the classification criteria are not met.*

### **SECTION 12: Ecological information**

- **12.1 Toxicity**
- **Aquatic toxicity:** *No further relevant information available.*
- **12.2 Persistence and degradability** *No further relevant information available.*
- **12.3 Bioaccumulative potential** *No further relevant information available.*
- **12.4 Mobility in soil** *No further relevant information available.*
- **Additional ecological information:**
- **General notes:**  
*Water hazard class 1 (German Regulation) (Self-assessment): slightly hazardous for water  
Do not allow undiluted product or large quantities of it to reach ground water, water course or sewage system.*
- **12.5 Results of PBT and vPvB assessment**
- **PBT:** *Not applicable.*
- **vPvB:** *Not applicable.*
- **12.6 Other adverse effects** *No further relevant information available.*

### **SECTION 13: Disposal considerations**

- **13.1 Waste treatment methods**
- **Recommendation** *Smaller quantities can be disposed of with household waste.*
- **Uncleaned packaging:**
- **Recommendation:** *Disposal must be made according to official regulations.*
- **Recommended cleansing agents:** *Water, if necessary together with cleansing agents.*

### **SECTION 14: Transport information**

- **14.1 UN-Number**
- **ADR, ADN, IMDG, IATA** Void

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*Trade name: Magnesium carbonate*

(Contd. of page 4)

- 14.2 UN proper shipping name - ADR, ADN, IMDG, IATA	Void
- 14.3 Transport hazard class(es) - ADR, ADN, IMDG, IATA - Class	Void
- 14.4 Packing group - ADR, IMDG, IATA	Void
- 14.5 Environmental hazards: - Marine pollutant:	No
- 14.6 Special precautions for user	Not applicable.
- 14.7 Transport in bulk according to Annex II of Marpol and the IBC Code	Not applicable.
- Transport/Additional information:	Not dangerous according to the above specifications.
- UN "Model Regulation":	Void

### SECTION 15: Regulatory information

- 15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture
- TSCA (Toxic Substances Control Act): ACTIVE
- TSCA new (21st Century Act) ACTIVE/EXEMPT
- Philippines Inventory of Chemicals and Chemical Substances Substance is not listed.
- Chinese Chemical Inventory of Existing Chemical Substances Substance is not listed.
- Australian Inventory of Chemical Substances Substance is not listed.
- Korean Existing Chemical Inventory KE-22686
- New Zealand Inventory of Chemicals Substance is listed.
- HSNO Approval numbers Substance is not listed.
- TCSI - Taiwan Chemical Substance Inventory Substance is listed.
- Japanese Existing Chemical Substances 1-155
- PRTR Class 1 designated substances Substance is not listed.
- PRTR Class 2 designated substances Substance is not listed.
- Directive 2012/18/EU
- Named dangerous substances - ANNEX I Substance is not listed.
- Regulation (EU) No 649/2012 Substance is not listed.
- EINECS Substance is listed.
- REACH - Pre-registered substances Substance is not listed.
- 15.2 Chemical safety assessment: A Chemical Safety Assessment has been carried out.

### SECTION 16: Other information

*This safety data sheet contains only safety-relevant information. The information is based on the state of our knowledge at the time of revision, but does not constitute an assurance of product properties, product information or product specifications and does not establish a contractual legal relationship.*

*This document is only valid in its unchanged form. Dr. Paul Lohmann® accepts no responsibility for the form and content of any changes made by third parties, nor for any resulting damage or claims.*

*The information is not transferable to other products. Insofar as the product named in this safety data sheet is mixed, blended, processed with or without other materials, the information in this safety data sheet cannot be transferred to the new material produced in this way, unless expressly stated otherwise.*

*The data sheet does not release the user from the obligation to ensure that he acts in accordance with all regulations in connection with his activity.*

- Department issuing SDS: Product safety department

(Contd. on page 6)

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<b>Trade name: Magnesium carbonate</b>	
<p><b>- Contact:</b></p> <p><b>- Abbreviations and acronyms:</b>  <i>RID: Règlement international concernant le transport des marchandises dangereuses par chemin de fer (Regulations Concerning the International Transport of Dangerous Goods by Rail)</i>  <i>ICAO: International Civil Aviation Organisation</i>  <i>ADR: Accord européen sur le transport des marchandises dangereuses par Route (European Agreement concerning the International Carriage of Dangerous Goods by Road)</i>  <i>IMDG: International Maritime Code for Dangerous Goods</i>  <i>IATA: International Air Transport Association</i>  <i>GHS: Globally Harmonised System of Classification and Labelling of Chemicals</i>  <i>EINECS: European Inventory of Existing Commercial Chemical Substances</i>  <i>CAS: Chemical Abstracts Service (division of the American Chemical Society)</i>  <i>PBT: Persistent, Bioaccumulative and Toxic</i>  <i>vPvB: very Persistent and very Bioaccumulative</i>  <i>PRTR: Law concerning Pollutant Release and Transfer Register</i></p> <p><b>- * Data compared to the previous version altered.</b></p>	(Contd. of page 5)

Item B.11

**Substance report from the National Institute of Environmental Health Sciences**

<ul style="list-style-type: none"> <li>The National Institute of Environmental Health Sciences (NIEHS)/ The National Toxicology Program (NTP)  <a href="https://www.niehs.nih.gov/">https://www.niehs.nih.gov/</a></li> </ul>	Information does not exist
<ul style="list-style-type: none"> <li>The Agency for Toxic Substances and Disease Registry (ATSDR):  <a href="https://www.atsdr.cdc.gov/">https://www.atsdr.cdc.gov/</a></li> </ul>	Non applicable
<ul style="list-style-type: none"> <li>U.S. National Library of Medicine's PubChem database:  <a href="https://pubchem.ncbi.nlm.nih.gov/">https://pubchem.ncbi.nlm.nih.gov/</a></li> </ul>	<a href="https://pubchem.ncbi.nlm.nih.gov/#query=546-93-0">https://pubchem.ncbi.nlm.nih.gov/#query=546-93-0</a>
<p> <b>MAGNESIUM CARBONATE; 546-93-0; Magnesite; Carbonic Acid, Magnesium Salt; 13717-00-5; Carbonic Acid, Magnesium Salt (1:1); Magnesium;Carbonate; Magnesite Dust; ...</b></p> <p>Compound CID: 11029        MF: <chem>MgO3</chem> MW: 84.31g/mol        IUPAC Name: magnesium;carbonate        Isomeric SMILES: C(=O)[O-].[Mg+2]        InChIKey: ZLNQNXFFQJAJD-UHFFFAOYSA-L        InChI: InChI=1S/CH2O3.Mg/c2-1(3)4;/h(2,2,3,4);/q;+2/p-2        Create Date: 2005-06-24</p>	
<ul style="list-style-type: none"> <li>The Occupational Safety and Health Administration (OSHA):  <a href="https://www.osha.gov/">https://www.osha.gov/</a></li> </ul>	<p><a href="https://www.osha.gov/sic-manual/2819">https://www.osha.gov/sic-manual/2819</a></p> <p><i>Description for 2819: Industrial Inorganic Chemicals, Not Elsewhere Classified =&gt; "magnesium carbonate" is listed :        "Establishments primarily engaged in manufacturing industrial inorganic chemicals, not elsewhere classified. Establishments primarily engaged in mining, milling, or otherwise preparing natural potassium, sodium, or boron compounds (other than common salt) are classified in Industry 1474. Establishments primarily engaged in manufacturing household bleaches are classified in Industry 2842; those manufacturing phosphoric acid are classified in Industry 2874; and those manufacturing nitric acid, anhydrous ammonia, and other nitrogenous fertilizer materials are classified in Industry 2873"</i></p> <p><a href="https://www.osha.gov/chemicaldata/403">https://www.osha.gov/chemicaldata/403</a></p> <p>Refer to Appendix 1 :</p> <p>        MAGNESITE _        Occupational Safety a</p> <p>Appendix 2 :</p> <p>        MAGNESITE _        CAMEO Chemicals _ N</p>

<ul style="list-style-type: none"> <li>The National Institute for Occupational Safety and Health (NIOSH): <a href="https://www.cdc.gov/niosh/index.htm">https://www.cdc.gov/niosh/index.htm</a></li> </ul>	<p>Refer to Appendix 3 :</p>  <p>CDC - NIOSH Pocket Guide to Chemical Ha</p>
<ul style="list-style-type: none"> <li>U.S. Environmental Protection Agency (U.S. EPA)</li> </ul>	<p>Not applicable</p>
<ul style="list-style-type: none"> <li>U.S. Food and Drug Administration (FDA)</li> </ul>	<p><a href="https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcr/CFRSearch.cfm?fr=184.1425&amp;SearchTerm=magnesium%20carbonate">https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfcr/CFRSearch.cfm?fr=184.1425&amp;SearchTerm=magnesium%20carbonate</a></p>
<p>[Code of Federal Regulations] [Title 21, Volume 3] [CITE: 21CFR184.1425]</p> <p style="text-align: center;">TITLE 21--FOOD AND DRUGS CHAPTER I--FOOD AND DRUG ADMINISTRATION DEPARTMENT OF HEALTH AND HUMAN SERVICES SUBCHAPTER B - FOOD FOR HUMAN CONSUMPTION (CONTINUED)</p> <p>PART 184 -- DIRECT FOOD SUBSTANCES AFFIRMED AS GENERALLY RECOGNIZED AS SAFE Subpart B - Listing of Specific Substances Affirmed as GRAS</p> <p>Sec. 184.1425 Magnesium carbonate.</p> <p>(a) <b>magnesium carbonate</b> (molecular formula approximately <math>MgCO_3 \cdot 4Mg(OH)_2 \cdot 5H_2O</math>, CAS Reg. No. 39409-82-0) is also known as <b>magnesium carbonate hydroxide</b>. It is a white powder formed either by adding an alkaline carbonate (such as sodium carbonate) to a solution of magnesium sulfate or by carbonation of a slurry of magnesium hydroxide followed by boiling of the resulting <b>magnesium carbonate</b> .</p> <p>(b) The ingredient meets the specifications of the Food Chemicals Codex, 3d Ed. (1981), p. 177, which is incorporated by reference. Copies are available from the National Academy Press, 2101 Constitution Ave. NW., Washington, DC 20418, or available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: <a href="http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html">http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html</a>.</p> <p>(c) In accordance with § 184.1(b) (1), the ingredient is used in food with no limitation other than current good manufacturing practice. The affirmation of this ingredient as generally recognized as safe (GRAS) as a direct human food ingredient is based upon the following current good manufacturing practice conditions of use:</p> <p>(1) The ingredient is used as an anticaking and free-flow agent as defined in § 170.3(o) (1) of this chapter; a flour treating agent as defined in § 170.3(o) (13) of this chapter; a lubricant and release agent as defined in § 170.3(o) (18) of this chapter; a nutrient supplement as defined in § 170.3(o) (20) of this chapter; a pH control agent as defined in § 170.3(o) (23) of this chapter; a processing aid as defined in § 170.3(o) (24) of this chapter; and a synergist as defined in § 170.3(o) (31) of this chapter.</p> <p>(2) The ingredient is used in foods at levels not to exceed current good manufacturing practice.</p> <p>(d) Prior sanctions for this ingredient different from the uses established in this section do not exist or have been waived.</p> <p>[50 FR 13558, Apr. 5, 1985; 50 FR 16080, Apr. 24, 1985]</p>	

**Item B.B.12**

<p><i>Substance research reviews and research bibliographies</i></p>
<p>Title : Analytical methods in food additives determination: Compounds with functional applications, Author : Fernanda C.O.L.Martins, Michelle A. Sentanin, Djenaine De Souza Journal : Food Chemistry, Volume 272, 30 January 2019, Pages 732-750 Refer to Appendix 4</p>

Item B.13

<i>Substance is petitioned for §205.605(b) (nonagricultural, synthetic)</i>	
<i>Why the synthetic substance is necessary for the production or handling of an organic product</i>	<p>Magnesium carbonate is used during processing of liquid chicory into soluble powder. The use of Magnesium carbonate as a processing aid in the production of soluble chicory extract improves the fluidity and therefore the flow of the powder. Magnesium carbonate is used as a processing aid : anti-caking agent.</p>
	<p>Since well before 2015, we use silicon dioxide (E551) which is an authorized substance in the manufacture of foodstuffs (conventional as well as organic). The E551 questions the French authorities as to its nanotechnological character or not.</p> <p>The substitution of E551 by E504(i) is authorized as an additive, but not as a processing aid. However, for the manufacture of dried foodstuffs in powder form and instant chicory extract in particular, having a powder flow facilitator as a technological aid (presence in the finished product in a technically unavoidable trace state and absence of technological effect) is indispensable during the transformation. Without this type of auxiliary, these foodstuffs could not be produced.</p> <p>We would therefore like E504(i) to be authorized again as an authorized processing aid in the manufacture of foodstuffs (conventional and organic).</p>
<p><i>Nonsynthetic substances, synthetic substances on the National List that could be used in place of the petitioned synthetic substance</i></p> <p></p> <p>7 CFR 205.605 (up to date as of 8-26-2022)</p>	<p><a href="#">§ 205.605 Nonagricultural (nonorganic) substances allowed as ingredients in or on processed products labelled as “organic” or “made with organic (specified ingredients or food group(s)).”</a></p> <p>The following nonagricultural substances may be used as ingredients in or on processed products labelled as “organic” or “made with organic (specified ingredients or food group(s))” only in accordance with any restrictions specified in this section.</p> <p>(b) <i>Synthetics allowed:</i></p> <p>[...] Silicon dioxide - Permitted as a defoamer. Allowed for other uses when organic rice hulls are not commercially available.</p> <p><a href="#">[68 FR 61993]</a>, Oct. 31, 2003]</p>
<i>Beneficial effects to the environment, human health, or farm ecosystem from use of the synthetic substance that support its use instead of the use of a nonsynthetic substance or alternative cultural method.</i>	<p>Non applicable</p> <p>Not used during crop fields but after harvest, during the chicory transformation process.</p> <p>We would like Magnesium carbonate to be included in the list of substances authorized as a processing aid.</p> <p>We will not use Magnesium hydroxide carbonate in the chicory root cultivation phases but only during factory processing.</p>

## Appendix 1 : MAGNESITE \_ Occupational Safety and Health Administration

OSHA Occupational Chemical Database MAGNESITE

### MAGNESITE†

Chemical Identification	
CAS #	546-93-0; 13717-00-5
Formula	MgCO <sub>3</sub>
Synonyms	carbonate magnesium; hydromagnesite; magnesium carbonate; magnesium(II) carbonate

Physical Properties			
Physical description	White, odorless, crystalline powder.		
Boiling point	decomposes	Molecular weight	84.3
Freezing point/melting point	662°F (decomposes)	Vapor pressure	0 mmHg (approx)
Flash point		Vapor density	
Specific gravity	2.96	Ionization potential	
Lower explosive limit (LEL)		Upper explosive limit (UEL)	
NFPA health rating		NFPA fire rating	
NFPA reactivity rating		NFPA special instruction	
Vapor hazard ratio (VHR)			
Historical exceedance percentage			
Target organs			

Monitoring Methods Used by OSHA			
Analyte code (IMIS no.)	1615 (total); request using analyte code 9135	M113 (resp); request using analyte code 9130	
Sampling group			
Sampler/Sampling media	Tared low-ash 37 mm PVC filter, 5 microns [SLTC108]	Tared low-ash 37 mm PVC filter, 5 microns, preceded by 10 mm nylon Dorr-Oliver cyclone for respirable samples. [SLTC108]	

Monitoring Methods Used by OSHA			
Sampling time*	240-480 min	240-480 min	
Sampling volume (TWA)*	960 L	816 L	
Sampling flow rate (TWA)*	2 L/min	1.7 L/min ±5%	
Sampling volume (STEL/Peak/C)*			
Sampling flow rate (STEL/Peak/C)*			
Analytical method instruments	Gravimetric	Gravimetric	
Method reference	OSHA PV2121 (partially validated)	OSHA PV2121 (partially validated)	
Notes	Samples are analyzed gravimetrically. Results will be qualified based on the assumption that the material sampled is as identified by the compliance officer using available documentation of materials and processes; no further work on the sample will be performed due to method limitations.	Samples are analyzed gravimetrically. Results will be qualified based on the assumption that the material sampled is as identified by the compliance officer using available documentation of materials and processes; no further work on the sample will be performed due to method limitations. The standard applies to the Respirable Fraction.	
Special requirements	FOR TOTAL DUST SAMPLES, DO NOT USE A CYCLONE!!!	Samples must be collected with a Dorr-Oliver cyclone.	

\* All sampling instructions above are recommended guidelines for OSHA Compliance Safety and Health Officers (CSHOs), please see the corresponding OSHA method reference for complete details.

Wipe Method	
Sampler/Sampling media	

Bulk Method	

On-Site Screening Techniques				
Device				
Model/Type				
Sampling information (see manufacturer instructions)				

Exposure Limits							
OSHA PEL 8-hour TWA (ST) STEL (C) Ceiling Peak		NIOSH REL Up to 10-hour TWA (ST) STEL (C) Ceiling		ACGIH TLV® 8-hour TWA (ST) STEL (C) Ceiling		CAL/OSHA PEL 8-hour TWA (ST) STEL (C) Ceiling Peak	
PEL-TWA	15 mg/m <sup>3</sup> (total dust), 5 mg/m <sup>3</sup> (respirable fraction)	REL-TWA	10 mg/m <sup>3</sup> (total), 5 mg/m <sup>3</sup> (resp)	TLV-TWA		PEL-TWA	10 mg/m <sup>3</sup> (total dust), 5 mg/m <sup>3</sup> (respirable fraction)
PEL-STEL		REL-STEL		TLV-STEL		PEL-STEL	
PEL-C		REL-C		TLV-C		PEL-C	
Skin notation	N	Skin notation	N	Skin notation	N	Skin notation	N
<b>Notes:</b> See 29 CFR 1910.1000 Table Z-1.		<b>Notes:</b>		<b>Notes:</b> See Appendix B: Particles (insoluble or poorly soluble) Not Otherwise Specified (PNOS)		<b>Notes:</b> as Particulates not otherwise regulated. See also footnote [n]	
<b>Health factors:</b> See NIH-NLM PubChem.		IDLH					
<b>Carcinogenic classifications:</b> Not listed		<b>Notes:</b>					
AIHA emergency response planning guidelines - ERPG-1/ERPG-2/ERPG-3:							

Additional Resources and Literature References	
NOAA: CAMEO Chemicals - <a href="#">Magnesite</a>	
NIOSH: Pocket Guide to Chemical Hazards - <a href="#">Magnesite</a>	
<b>Literature References</b>	
<ul style="list-style-type: none"> <li>ACGIH: <i>Documentation of the Threshold Limit Values (TLVs) and Biological Exposure Indices (BEIs)</i> - Appendix B: Particles (insoluble or poorly soluble) Not Otherwise Specified (PNOS). See annual publication for most recent information.</li> </ul>	

Last Updated Date : 01/04/2021

## Appendix 2 : MAGNESITE \_ CAMEO Chemicals \_ NOAA

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MAGNESITE | CAMEO Chemicals | NOAA



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### Chemical Datasheet

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## MAGNESITE

[Chemical Identifiers](#) | [Hazards](#) | [Response Recommendations](#) | [Physical Properties](#) | [Regulatory Information](#) | [Alternate Chemical Names](#)

### Chemical Identifiers

[What is this information?](#) ▶

**CAS Number**  
546-93-0

**UN/NA Number**  
none

**DOT Hazard Label**  
data unavailable

**USCG CHRIS Code**  
none

**NIOSH Pocket Guide**  
[Magnesite](#) ⓘ

**International Chem Safety Card**  
[MAGNESIUM CARBONATE](#) ⓘ

**NFPA 704**  
data unavailable

### General Description

White, yellowish, grayish-white or brown crystalline solid or crystalline powder. Density: 3-3.1 g cm-3. An important ore for magnesium. Used in the manufacture of materials capable of withstanding very high temperatures. Sometimes used to produce carbon dioxide.

### Hazards

[What is this information?](#) ▶

### Reactivity Alerts

none

### Air & Water Reactions

No rapid reaction with air. No rapid reaction with water.

https://cameochemicals.noaa.gov/chemical/25039

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MAGNESITE | CAMEO Chemicals | NOAA

### Fire Hazard

No information available.

### Health Hazard

Excerpt from [NIOSH Pocket Guide for Magnesite](#) ⓘ:

Exposure Routes: Inhalation, skin and/or eye contact

Symptoms: Irritation eyes, skin, respiratory system; cough

Target Organs: Eyes, skin, respiratory system (NIOSH, 2022)

### Reactivity Profile

MAGNESITE has generally low chemical reactivity. Non-flammable and non-combustible. Reacts with acids and acidic salts to generate gaseous carbon dioxide with effervescence (bubbling). The reaction may be rapid and exothermic with concentrated solutions of acids. The effervescence can create foaming. Incompatible with formaldehyde.

### Belongs to the Following Reactive Group(s)

- [Carbonate Salts](#)

### Potentially Incompatible Absorbents

No information available.

### Response Recommendations

[What is this information?](#) ▶

### Isolation and Evacuation

No information available.

### Firefighting

No information available.

### Non-Fire Response

No information available.

### Protective Clothing

Excerpt from [NIOSH Pocket Guide for Magnesite](#) ⓘ:

Skin: No recommendation is made specifying the need for personal protective equipment for the body.

https://cameochemicals.noaa.gov/chemical/25039

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MAGNESITE | CAMEO Chemicals | NOAA

Eyes: No recommendation is made specifying the need for eye protection.

Wash skin: No recommendation is made specifying the need for washing the substance from the skin (either immediately or at the end of the work shift).

Remove: No recommendation is made specifying the need for removing clothing that becomes wet or contaminated.

Change: No recommendation is made specifying the need for the worker to change clothing after the workshift. (NIOSH, 2022)

**DuPont Tychem® Suit Fabrics**

No information available.

**First Aid**

Excerpt from [NIOSH Pocket Guide for Magnesite](#) (2):

Eye: IRRIGATE IMMEDIATELY - If this chemical contacts the eyes, immediately wash (irrigate) the eyes with large amounts of water, occasionally lifting the lower and upper lids. Get medical attention immediately.

Breathing: FRESH AIR - If a person breathes large amounts of this chemical, move the exposed person to fresh air at once. Other measures are usually unnecessary. (NIOSH, 2022)

**Physical Properties**

[What is this information?](#)

**Chemical Formula:** MgCO3

Flash Point: data unavailable

Lower Explosive Limit (LEL): data unavailable

Upper Explosive Limit (UEL): data unavailable

Autoignition Temperature: data unavailable

**Melting Point:** 662°F (Decomposes) (NIOSH, 2022)

**Vapor Pressure:** 0 mmHg (approx) (NIOSH, 2022)

Vapor Density (Relative to Air): data unavailable

**Specific Gravity:** 2.96 (NIOSH, 2022)

**Boiling Point:** Decomposes (NIOSH, 2022)

**Molecular Weight:** 84.3 (NIOSH, 2022)

**Water Solubility:** 0.01 % (NIOSH, 2022)

Ionization Energy/Potential: data unavailable

<https://cameochemicals.noaa.gov/chemical/25039>

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MAGNESITE | CAMEO Chemicals | NOAA

IDLH: data unavailable

**AEGLs (Acute Exposure Guideline Levels)**

No AEGL information available.

**ERPGs (Emergency Response Planning Guidelines)**

No ERPG information available.

**PACs (Protective Action Criteria)**

Chemical	PAC-1	PAC-2	PAC-3
Magnesium carbonate; (Magnesite) (546-93-0)	45 mg/m3	260 mg/m3	1600 mg/m3

(DOE, 2018)

**Regulatory Information**

[What is this information?](#)

**EPA Consolidated List of Lists**

No regulatory information available.

**CISA Chemical Facility Anti-Terrorism Standards (CFATS)**

No regulatory information available.

**OSHA Process Safety Management (PSM) Standard List**

No regulatory information available.

**Alternate Chemical Names**

[What is this information?](#)

<https://cameochemicals.noaa.gov/chemical/25039>

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MAGNESITE | CAMEO Chemicals | NOAA

- APOLDA
- C.I. 77713
- CARBONATE MAGNESIUM
- DCI LIGHT MAGNESIUM CARBONATE
- DESTAB
- GOLD STAR
- GOLD STAR (CARBONATE)
- GP 20
- GP 20 (CARBONATE)
- GP 30
- GP 30 (CARBONATE)
- HYDROMAGNESITE
- KIMBOSHI
- MA 70 (CARBONATE)
- MAGFY
- MAGNESIA
- MAGNESITE
- MAGNESIUM CARBONATE
- MAGNESIUM CARBONATE (1:1)
- MAGNESIUM CARBONATE (MGCO3)
- MAGNESIUM CARBONATE; (MAGNESITE)
- MAGNESIUM(II) CARBONATE
- STAN-MAG MAGNESIUM CARBONATE

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Web site owner: [Office of Response and Restoration](#), [National Ocean Service](#), [National Oceanic and Atmospheric Administration](#) - [USA.gov](#).

CAMEO Chemicals version 2.8.0 rev 1.

## Appendix 3 : CDC-NIOSH Pocket

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CDC - NIOSH Pocket Guide to Chemical Hazards - Magnesite

Promoting productive workplaces  
through safety and health research / 

### Magnesite

#### Synonyms & Trade Names

Carbonate magnesium, Hydromagnesite, Magnesium carbonate, Magnesium(II) carbonate [Note: Magnesite is a naturally-occurring form of magnesium carbonate.]

#### CAS No.

546-93-0

#### RTECS No.

[OM2470000](#)

#### DOT ID & Guide

#### Formula

MgCO<sub>3</sub>

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CDC - NIOSH Pocket Guide to Chemical Hazards - Magnesite

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#### Physical Description

White, odorless, crystalline powder.

#### Molecular Weight

84.3

#### Boiling Point

Decomposes

#### Melting Point

662°F (Decomposes)

#### Solubility

0.01%

1

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CDC - NIOSH Pocket Guide to Chemical Hazards - Magnesite

**Lower Explosive Limit**

NA

Noncombustible Solid

**Incompatibilities & Reactivities**

Acids, formaldehyde

**Exposure Routes**

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CDC - NIOSH Pocket Guide to Chemical Hazards - Magnesite

**Respirator Recommendations**

Not available.

[Important additional information about respirator selection](#)

**See also**

[INTRODUCTION](#), ICSC CARD: 0969

## Appendix 4 : Publication “Analytical methods in food additives determination: Compounds with functional applications”

Food Chemistry 272 (2019) 732–750



Contents lists available at ScienceDirect

Food Chemistry

journal homepage: [www.elsevier.com/locate/foodchem](http://www.elsevier.com/locate/foodchem)



Review

### Analytical methods in food additives determination: Compounds with functional applications



Fernanda C.O.L. Martins<sup>a</sup>, Michelle A. Sentanin<sup>b</sup>, Djenaine De Souza<sup>a,\*</sup>

<sup>a</sup> Laboratório de Eletroanálise Aplicada a Biotecnologia e a Engenharia de Alimentos – LEABE, Campus de Patos de Minas, Instituto de Química, Universidade Federal de Uberlândia, Av. Getúlio Vargas, 230, Centro 38700-126, Patos de Minas-MG, Brazil

<sup>b</sup> Laboratório de Análise e Química de Alimentos, Campus de Patos de Minas, Faculdade de Engenharia Química, Universidade Federal de Uberlândia, Av. Getúlio Vargas, 230, Centro 38700-126, Patos de Minas-MG, Brazil

#### ARTICLE INFO

**Keywords:**  
 Food additives  
 Analytical methods  
 Spectroscopy  
 Chromatography  
 Electroanalysis  
 Food control

#### ABSTRACT

This work describes the 25 classes of food additives that contain about 230 compounds with technological, sensorial and/or microbiological functionalities. These compounds are added to foods at the processing, packaging and transport steps, to improve the standard of quality, durability and stability of the product and adjust the colour, smell and flavour attributes. Food industries need to follow national and international quality standards that establish the conditions under which food additives may be used, and the food safety is guaranteed by strict quality control. This review describes the analytical techniques used in identification and quantification of food additives in foodstuffs, highlighting the main characteristics of each method (spectroscopy, chromatography and electroanalysis), and indicating the advantages and disadvantages typical of the methods used. Perspectives of the chemical analysis in the food industries are also discussed.

#### 1. Introduction

In the last decades, the increased population and lifestyle changes promoted considerable alterations in the formulation of food products. Additionally, the transformation of the eating habits and modification in the nutritional demands caused an important transformation in the food industries, which involved the incorporation of additional food ingredients to food products. The purpose of these compounds (food additives) is to add dietary nutrition (fortifiers) (Martins, Franco, Muñoz, & De Souza, 2017), increase the shelf life and/or to improve the physicochemical, sensorial and microbiological properties of the industrialised foods (Damodaran & Parkin, 2017).

A vast variety and quantity of chemical additives are employed in industrialised foods, to maintain and/or improve the biological, physicochemical, rheological and sensorial properties, such as pH, texture, homogeneity, colour, flavour, sweetness, crunchiness, overall quality and stability, and to extend the expiry date. A plethora of compounds presenting specific functionality is used to achieve these objectives, promoting differentiated quality in industrialised foods. The food industries employ about 25 classes of food additives, which are used according to the specific legislation of each country and following a food safety policy, based on the *Codex Alimentarius* (Food and Agriculture Organisation of the United Nations/World Health Organisation [FAO/WHO], 2016).

For all approved food additives, the legislation should indicate the foods to which the food additive may be added, the conditions under which the food additive may be used and recommend the maximum permissible quantity of the additive, which is based on a level that assures consumer safety. For this, the industries and the regulatory agencies require rigorous quality control in the identification of the class of the food additives and the level of quantification at which they are employed. Ensuring that any illegal additives are avoided, also demands rigorous evaluation.

The current review describes the different classes of the food additives and the main analytical methods employed in the analysis of these compounds, indicating the advantages, disadvantages and perspectives of the chemical analysis in the food industry.

#### 2. Food additives

According to the *Codex Alimentarius* (FAO/WHO, 2016), which is recognised as the international standard, food additives are any compound not typically consumed as a food by itself and not normally used as an ingredient in the food but is intentionally added in the manufacture, processing, preparation, treatment, packing, packaging, transport and holding of the food, to perform a technological function (including organoleptic). The term does not include additives or

\* Corresponding author.

E-mail addresses: [michelle@ufu.br](mailto:michelle@ufu.br) (M.A. Sentanin), [djenaine@ufu.br](mailto:djenaine@ufu.br) (D. De Souza).

<https://doi.org/10.1016/j.foodchem.2018.08.060>

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Available online 23 August 2018

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contaminants added to food for preserving or enhancing the nutritional properties. The use of food additives improves the quality of the final product and increases the shelf life of the foodstuffs on retail shelves.

In general, the food industry can only use food additives that offer a reasonable technological need that cannot be achieved by other economically and technically viable methods. In addition, these compounds cannot present a risk to the health of consumers, at the level of use proposed. Thus, the FAO/WHO (2017), by the implementation of the *Codex Alimentarius*, and the European Union (EU, 2017) establish the conditions under which food additives may be used by the food industries.

The well-developed food safety policies established by the FAO (2016) and EU (2017) are the point of reference for other official food authorities around the world and the creation of specific legislation in other countries. In Brazil, the National Sanitary Vigilance Agency (ANVISA) is responsible for the authorisation of the use and inspection of food additives in the industries (ANVISA, 2018). Furthermore, industries that export industrialised foods need to follow national and international quality standards, according to regulations and laws, and adequately meet the consumers' expectations, by employing an adequate amount of the food additive.

### 2.1. Food additives classification

The FAO and EU employ the food additives classification, according to the food type in which they may be added. The food type categories include, for example, dairy and dairy-based products, fats and oils, edible ices, fruits and vegetables, confectionery, cereal and cereal products, eggs and eggs products, sweeteners, salts, spices, soups, sauces, salads, protein products, foodstuffs intended for nutritional uses, beverages, ready-to-eat savouries and prepared foods (FAO/WHO, 2016).

According to the industrial use, the food additives are classified into 25 classes, which include about 230 different compounds. Some compounds present more than one principal action, due to their different functionalities, which are dependent on the added quantities and processing mode of the food. Fig. 1 shows the relative percentage of each food additive, considering the functional classes.

The main technological functions of food additives involve pH control, viscosity, stability and homogeneity; the biological functions are related to stability, degradation process inhibition and shelf life; and/or the sensorial functions include, among others, colour, smell and flavour. The concentration of the incorporated additive, the step at which it is added, the mode in which it is added and the food type, can be adjusted, according to the desired functionalities. Table 1 shows the function and uses of the respective food additives, as described above.

#### 2.1.1. Acidity regulators

The acidity regulators are substances capable of controlling or modifying the pH of foodstuffs, thereby influencing the smell, flavour, viscosity, texture and, mainly, shelf life, by directly impacting on the oxidation/enzymatic reactions and inhibition of microbiological growth. These compounds are constituted by organic acids, inorganic acids, bases and conjugated salts, and can act alone or in conjunction with the buffering system (Belitz, Groch, & Schieberle, 2009).

The organic acids mainly used as acidity regulators are acetic, citric, malic, benzoic and formic. These acids act as food preservatives, chelating or sequestrant agents, in the setting of pectin gels, emulsifiers, sterilisers, anti-foamers and flavouring agents. The most common basic acidity regulators are salts of the phosphates, lactates and citrates, employed for pH stabilisation, producing a buffering in the foodstuffs system, colour retention and complexation reactions of metals, to improve the sensorial quality of the foodstuffs. Strong inorganic acids and bases are also used in the processing of vegetables and fruits (Msagati, 2012).

Sometimes the pH must be alternated during the industrial process, by using a buffer system, according to the desired pH values. The most common buffer systems employed are citric acid/sodium citrate (pH 2.1–4.7), acetic acid/sodium acetate (pH 3.6–5.6) and ortho- and pyrophosphate anions (pH 2.0–12.0). These buffers aid in pH control for protein stabilisation, without causing alterations to the smell and flavour of the food (Danodaran & Parkin, 2017).

#### 2.1.2. Anticaking agents

In powdered foods, agglutination and lump formation can occur, which decrease the quality and interfere in the solubility and sensorial

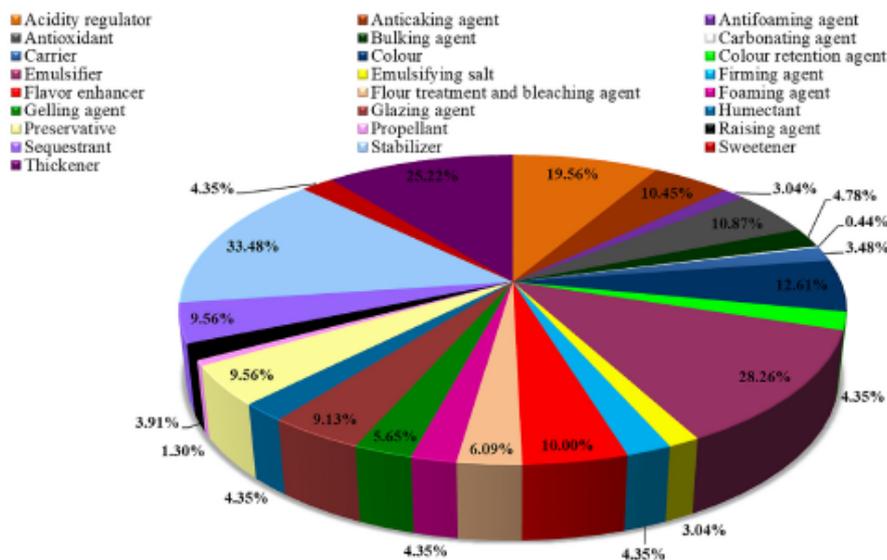


Fig. 1. Perceptual relationship of each class of the food additives use in food industries.

**Table 1**  
 Chemical additives used in food industries according main technological functions (FDA-U.S. Food Administ., 2018); (Food and Agriculture Organization of the United Nations World Health Organization, 2016).

Class	Additives
1 Acidity regulator	Glacial acetic acid; Adipates; Aluminum ammonium sulfate; Ammonium carbonate; Ammonium hydrogen carbonate; Ammonium hydroxide; Ascorbic acid, L; Calcium carbonate; Calcium hydroxide; Calcium lactate; Calcium oxide; Citric Acid; Fumaric Acid; Glucono delta-lactone; Hydrochloric acid; Lactic acid, L-, D- and DL; Magnesium Carbonate; Magnesium hydroxide; Magnesium hydroxide carbonate; Magnesium oxide; Malic acid, DL; Phosphoric acid; Sodium dihydrogen phosphate; Trisodium phosphate; Phosphates; Potassium acetate; Potassium carbonate; Potassium dihydrogen citrate; Potassium hydrogen carbonate; Potassium hydroxide; Potassium lactate; Sodium acetate; Sodium aluminum phosphates; Sodium carbonate; Sodium diacetate; Sodium dihydrogen citrate; Sodium DL-malate; Sodium fumarate; Sodium hydrogen carbonate; Sodium hydroxide; Sodium lactate; Sodium sesquicarbonate; Tartarates; Tricalcium citrate; Tripotassium citrate; Trisodium citrate;
2 Anticaking agent	Calcium silicate; Carnauba wax; Castor oil; Ferric ammonium citrate; Ferrocyanides; Magnesium hydroxide carbonate; Magnesium oxide; Magnesium silicate, synthetic; Mannitol; Microcrystalline cellulose (cellulose gel); Phosphates; Polydimethylsiloxane; Powdered cellulose; Salts of myristic, palmitic and stearic acids with ammonia, calcium, potassium and sodium; Salts of oleic acid with calcium, potassium and sodium; Silicon dioxide, amorphous; Sodium aluminosilicate; Sodium carbonate; Sodium hydrogen carbonate; Sodium sesquicarbonate; Talk;
3 Antifoaming agent	Calcium alginate; Microcrystalline wax; Mono- and di-glycerides of fatty acids; Polydimethylsiloxane; Polyethylene glycol; Polysorbates; Silicon dioxide, amorphous;
4 Antioxidant	Ascorbic acid, L; Ascorbyl esters; Butylated hydroxyanisole; Butylated hydroxytoluene; Calcium ascorbate; Citric Acid; Citric and fatty acid esters of glycerol; Erythorbic acid (isoascorbic acid); Ethylene diamine tetra acetates; Guaiacresin; Isopropyl citrates; Nitrous oxide; Phosphoric acid; Potassium lactate; Propyl gallate; Sodium ascorbate; Sodium erythorbate (sodium isoascorbate); Sodium lactate; Stannous chloride; Stearyl citrate; Sulfites; Tartrates; Tertiary butylhydroquinone; Thioldipropionates; Tocopherols;
5 Bulking agent	Hydroxypropyl methyl cellulose; Mannitol; Methyl cellulose; Microcrystalline cellulose (cellulose gel); Polydextroses; Powdered cellulose; Processed Eucheama sea weed (PES); Propylene glycol alginate; Sodium alginate; Sodium carboxymethyl cellulose (cellulose gum); Sodium lactate; Carbon dioxide;
6 Carbonating agent	
7 Carrier	Castor oil; Cyclodextrin, Beta-; Dextrins, roasted starch; Gum Arabic (Acacia gum); Magnesium hydroxide carbonate; Polyethylene glycol; Processed Eucheama sea weed (PES); Silicon dioxide, amorphous;
8 Colour	Allura red AC; Amaranth; Annatto extracts, Beta-based; Annatto extracts, Norbixin-based; Azorubine (Garmoisine); Brilliant black (Black PN); Brilliant blue FCF; Brown HF; Canthaxanthin; Caramel I – Plain caramel; Caramel II – Sulfite caramel; Caramel III – Ammonia caramel; Caramel IV – Sulfite ammonia caramel; Carmine; Carotenes, Beta-, vegetable; Carotenoids; Chlorophylls and Chlorophyllins, copper complexes; Curcumin; Erythrosine; Fast green FCF; Grape skin extract; Indigotine (Indigo Carmine); Iron oxides; Lutein from <i>Tagetes erecta</i> ; Ponceau 4R (Cochineal red A); Riboflavin; Sunset yellow FCF; Tartrazine; Zeaxanthin, synthetic;
9 Colour retention agent	Aluminum ammonium sulfate; Ethylene diamine tetra acetates; Ferrous gluconate; ferrous lactate; Magnesium Carbonate; Magnesium chloride; Magnesium hydroxide; Magnesium hydroxide carbonate; Nitrites; Stannous chloride;
10 Emulsifier	Acetic and fatty acid esters of glycerol; Agar; Alginate acid; Ammonium alginate; Ammonium salts of phosphoric acid; Beeswax; Candelilla wax; Carob bean gum; Carrageenan; Castor oil; Citric and fatty acid esters of glycerol; Dextrins, roasted starch; Diacetyltartaric and fatty acid esters of glycerol; Dioctyl sodium sulfosuccinate; Glycerol ester of wood rosin; Guar gum; Gum Arabic (Acacia gum); Hydroxypropyl cellulose; Hydroxypropyl starch; Karaya gum; Konjac flour; Lactic and fatty acid esters of glycerol; Lecithin; Methyl cellulose; Methyl ethyl cellulose; Microcrystalline cellulose (cellulose gel); Mono- and di-glycerides of fatty acids; Monostarch phosphate; Oxidized starch; Peetins; Phosphated distarch phosphate; Sodium dihydrogen phosphate; Trisodium phosphate; Phosphates; Polyethylene glycol; Polyglycerol esters of fatty acids; Polyglycerol esters of interesterified ricinoleic acid; Polyoxyethylene stearates; Polysorbates; Polyvinylpyrrolidone; Potassium alginate; Powdered cellulose; Processed Eucheama sea weed (PES); Propylene glycol; Propylene glycol alginate; Propylene glycol esters of fatty acids; Salts of myristic, palmitic and stearic acids with ammonia, calcium, potassium and sodium; Salts of oleic acid with calcium, potassium and sodium; Sodium alginate; Sodium aluminum phosphates; Sodium dihydrogen citrate; Sodium lactate; Sorbitan esters of fatty acids; Starch acetate; Starch sodium octenyl succinate; Starches, enzyme treated; Stearoyl lactylates; Stearyl citrate; Sucroglycerides; Sucrose acetate isobutyrate; Sucrose esters of fatty acids; Sucrose oligoesters, type I and type II; Tragacanth gum; Trisodium citrate; Xanthan gum;
11 Emulsifying salt	Sodium dihydrogen phosphate; Phosphates; Potassium dihydrogen citrate; Sodium aluminum phosphates; Sodium lactate; Tripotassium citrate; Trisodium citrate;
12 Firming agent	Aluminum ammonium sulfate; Calcium chloride; Calcium hydroxide; Calcium lactate; Calcium sulfate; Curdlan; Magnesium sulfate; Phosphates; Potassium chloride; Tricalcium citrate;
13 Flavor enhancer	Benzoyl peroxide; Calcium 5'-guanylate; Calcium 5'-inosinate; Calcium 5'-ribonucleotides; Calcium di-L-glutamate; Dipotassium 5'-guanylate; Disodium 5'-guanylate; Disodium 5'-inosinate; Disodium 5'-ribonucleotides; Ethyl maltol; Glutamic acid, L (+); Guanylic acid, 5'; Inosinic acid, 5'; Magnesium di-L-glutamate; Magnesium sulfate; Maltol; Monoammonium L-glutamate; Monopotassium L-glutamate; Monosodium L-glutamate; Neotame; Potassium 5'-inosinate; Sulfites; Tartrates;
14 Flour treatment agent	Alpha amylase from <i>Aspergillus oryzae</i> var.; Alpha amylase from <i>Bacillus subtilis</i> ; Azodicarbonamide; Benzoyl peroxide; Bromelain; Calcium lactate; Calcium oxide; Calcium sulfate; Carbohydrase from <i>Bacillus licheniformis</i> ; Chlorine; Citric and fatty acid esters of glycerol; Phosphates; Polysorbates; Sulfites;
15 Foaming agent	Ammonium alginate; Calcium alginate; Carbon dioxide; Methyl ethyl cellulose; Microcrystalline cellulose (cellulose gel); Nitrogen; Nitrous oxide; Polysorbates; Sucrose esters of fatty acids; Xanthan gum;
16 Gelling agent	Agar; Alginate acid; Ammonium alginate; Calcium alginate; Carrageenan; Curdlan; Konjac flour; Peetins; Potassium alginate; Processed Eucheama sea weed (PES); Sodium alginate; Sodium carboxymethyl cellulose (cellulose gum); Tara gum;
17 Glazing agent	Beeswax; Candelilla wax; Carnauba wax; Carrageenan; Castor oil; Gum Arabic (Acacia gum); Hydrogenated poly-1-decenes; Konjac flour; Microcrystalline wax; Mineral oil, high viscosity; Mineral oil, medium viscosity; Polyethylene glycol; Polyvinyl alcohol; Polyvinylpyrrolidone; Processed Eucheama sea weed (PES); Propylene glycol; Pullulan; Shellac, bleached; Sucrose esters of fatty acids; Sucrose oligoesters, type I and type II; Talk;
18 Humectant	Glycerol; Mannitol; Sodium dihydrogen phosphate; Trisodium phosphate; Phosphates; Polydextroses; Processed Eucheama sea weed (PES); Propylene glycol; Sodium DL-malate; Sodium lactate;
19 Preservative	Benzoates, Calcium acetate; Calcium propionate; Carbon dioxide; Dimethyl dicarbonate; Ethylene diamine tetra acetates; Hexamethylene tetramine; Hydroxybenzoates, para; Isopropyl citrates; Lauric arginate ethyl ester; Lysozyme; Natamycin (Pimaricin); Nisin; Nitrites; Ortho-phenylphenols; Trisodium phosphate; Potassium acetate; Propionic acid; Sodium acetate; Sodium diacetate; Sorbates; Sulfites;
20 Propellant	Carbon dioxide; Nitrogen; Nitrous oxide;
21 Raising agent	Ammonium carbonate; Ammonium hydrogen carbonate; Glucono delta-lactone; Sodium dihydrogen phosphate; Phosphates; Sodium aluminum phosphates; Sodium carbonate; Sodium hydrogen carbonate; Sodium sesquicarbonate;
22 Sequestrant	Calcium sulfate; Citric Acid; Citric and fatty acid esters of glycerol; Diacetyltartaric and fatty acid esters of glycerol; Ethylene diamine tetra acetates; Glucono delta-lactone; Isopropyl citrates; Lactic and fatty acid esters of glycerol; Phosphoric acid; Sodium dihydrogen phosphate; Trisodium phosphate; Potassium dihydrogen citrate; Sodium acetate; Sodium diacetate; Sodium dihydrogen citrate; Sodium gluconate; Stearyl citrate; Tartrates; Tricalcium citrate; Triethyl citrate; Tri-potassium citrate; Trisodium citrate;
23 Stabilizer	

(continued on next page)

Table 1 (continued)

Class	Additives
	Acetylated distarch adipate; Acetylated distarch phosphate; Acetylated oxidized starch; Acid-treated starch; Agar; Alginate; Alkaline treated starch; Aluminium ammonium sulfate; Ammonium alginate; Beeswax; Bleached starch; Calcium alginate; Calcium chloride; Carob bean gum; Citric and fatty acid esters of glycerol; Curdlan; Cyclodextrin, Beta-; Dextrins, roasted starch; Diacetyltartaric and fatty acid esters of glycerol; Ethylene diamine tetra acetate; Glycerol ester of wood rosin; Guar gum; Gum Arabic (Acacia gum); Hydroxypropyl cellulose; Hydroxypropyl distarch phosphate; Hydroxypropyl methyl cellulose; Hydroxypropyl starch; Karaya gum; Konjac flour; Lactic and fatty acid esters of glycerol; Magnesium chloride; Mannitol; Methyl cellulose; Methyl ethyl cellulose; Microcrystalline cellulose (cellulose gel); Mono- and di-glycerides of fatty acid; Mono starch phosphate; Oxidized starch; Pectins; Phosphated distarch phosphate; Sodium dihydrogen phosphate; Trisodium phosphate; Phosphates; Polydextroses; Polyglycerol esters of fatty acids; Polysorbates; Polyvinylpyrrolidone; Potassium alginate; Potassium carbonate; Potassium chloride; Potassium dihydrogen citrate; Potassium hydrogen carbonate; Powdered cellulose; Processed Euchema sea weed (PES); Propylene glycol alginate; Salts of myristic, palmitic and stearic acids with ammonia, calcium, potassium and sodium; Salts of oleic acid with calcium, potassium and sodium; Sodium alginate; Sodium aluminium phosphates; Sodium carbonate; Sodium carboxymethyl cellulose (cellulose gum); Sodium dihydrogen citrate; Sodium dihydrogen carbonate; Sorbitan esters of fatty acids; Starch acetate; Starch sodium octenyl succinate; Starches, enzyme treated; Stearoyl lactylates; Sucrose acetate isobutyrate; Sucrose esters of fatty acids; Sucrose oligoesters, type I and type II; Tara gum; Tartrates; Tragacanth gum; Triethyl citrate; Tripotassium citrate; Trisodium citrate; Xanthan gum;
24 Sweetener	Acesulfame potassium; Allitame; Aspartame; Aspartame-acesulfame salt; Cyclamates; Mannitol; Neotame; Saccharin; Steviol glycosides; Sucralose (Trichlorogalactosucrose)
25 Thickener	Acetylated distarch adipate; Acetylated distarch phosphate; Acetylated oxidized starch; Acid-treated starch; Agar; Alginate; Alkaline treated starch; Ammonium alginate; Bleached starch; Calcium alginate; Gandlella wax; Carob bean gum; Carrageenan; Curdlan; Cyclodextrin, Beta-; Dextrins, roasted starch; Distarch phosphate; Glycerol; Guar gum; Gum Arabic (Acacia gum); Hydroxypropyl cellulose; Hydroxypropyl distarch phosphate; Hydroxypropyl methyl cellulose; Hydroxypropyl starch; Karaya gum; Konjac flour; Mannitol; Methyl cellulose; Methyl ethyl cellulose; Microcrystalline cellulose (cellulose gel); Monostarch phosphate; Oxidized starch; Pectins; Phosphated distarch phosphate; Sodium dihydrogen phosphate; Trisodium phosphate; Phosphates; Polydextroses; Polyethylene glycol; Polyvinyl alcohol; Polyvinylpyrrolidone; Potassium alginate; Potassium chloride; Powdered cellulose; Processed Euchema sea weed (PES); Propylene glycol alginate; Pullulan; Sodium alginate; Sodium aluminium phosphates; Sodium carboxymethyl cellulose (cellulose gum); Sodium lactate; Starch acetate; Starch sodium octenyl succinate; Starches, enzyme treated; Talc; Tara gum; Tragacanth gum; Xanthan gum;

properties. Moreover, some food and food ingredients in powder form can absorb moisture readily when exposed to relative humidity change, and become sticky, agglomerate and form lumps, potentially leading to problems in food processing and storage (Figura & Teixeira, 2007).

Consequently, the food industries use anticaking agents, which are added in the production of dried powdered foods, salt, sugar confections and whey cheese products, among others. The most common anticaking agents are calcium and magnesium carbonates, phosphates and silicates. These are high water-binding agents that prevent caking by selectively binding the water and, notably, do not interfere with the final appearance of foodstuffs (Blekas, 2016).

#### 2.1.3. Antifoaming agents

Fermentation processes can often present an undesirable foam appearance because of the presence of surfactants in the fermentation steps. Hence, the addition of antifoaming agents is necessary to produce a barrier that reduces the oxygen transfer rate and, consequently, decreases the foam production. In practice, the antifoaming agent destabilises the liquid film that covers the air bubble, displacing the substances from the surface, and thereby preventing the foam stability. Besides, the antifoaming agent can interact with the surface-active stabilisers, promoting the collapse of the liquid film and destabilising the foam, or by dilution of the antifoaming agent in the mixture (McClure, Lamy, Black, Kavanagh, & Barton, 2017).

The inadequate use of antifoaming agents decreases the shelf life of the foodstuffs so their use should be controlled. The fatty acid esters, polyesters and silicone-based oils/emulsions are the most common antifoaming agents applied to food products and beverages (Msagati, 2012).

#### 2.1.4. Antioxidants

Oxidation by molecular oxygen is the main degradation process in foodstuffs and occurs as a consequence of the removal of electrons from a substrate (typically, oxygen-, nitrogen- or sulphur-containing) to an atom of oxygen present in the moisture, which gives rise to free radicals. Free radicals are highly reactive towards other molecules, due to their unpaired electrons, thereby leading to chain reactions. These reactions promote loss of the nutritional value and produce an undesirable flavour and smell. The initial step of the reaction is thermodynamically unfavourable, so the production of the first few radicals

necessary to initiate the propagation step requires the presence of a catalyst (e.g., light, metal ions, heat). To avoid or decrease the velocity of these reactions, the food industries add antioxidants, which interact with the free radicals and oxygen, responsible for promoting oxidation reactions in vitamins, natural colours and lipid components (Damodaran & Parkin, 2017).

Some substances naturally found in food (tocopherol and carotene) present antioxidant activity and contribute health benefits to industrialised foodstuffs. All synthetic antioxidants present chemical structures similar to natural phenolic compounds, which participate in the oxidation reactions through resonance-stabilised free radical forms. The main synthetic compounds are butylated hydroxyanisole, butylated hydroxytoluene and tertiary butylhydroquinone (Belitz et al., 2009). However, such synthetic antioxidants are not preferred due to toxicological concerns. Thus, increasing interest is focussed on identifying plant extracts as sources of antioxidants.

The application of antioxidants in foodstuffs increases the shelf life. However, their use must be carefully assessed and monitored, due to toxicity or inadequate concentrations, resulting in the formation of toxic compounds in foods that can be absorbed by the consumer and may cause health issues, such as degenerative diseases and mutations (Shibamoto & Bjeldanes, 2004). Furthermore, the inappropriate use of antioxidants can provoke unpleasant alterations in the flavour and aroma of the foodstuffs. The antioxidant efficiency is increased by the synergism among antioxidants; two or more antioxidants with different mechanisms (e.g., a combination of metal chelators and free radical scavengers), two or more free radical scavengers, whereby one of these substances is regenerated by others (e.g., the ascorbic acid/tocopherols system), protective action of one antioxidant that undergoes oxidation to shield the more effective antioxidant against oxidation (the interaction between carotenoids and tocopherols partly results from this mechanism) (Msagati, 2012). Table 1 lists the main antioxidants used by the food industries.

#### 2.1.5. Bulking agents

The volume is an important characteristic of foodstuffs since it contributes to a fundamental sensorial attribute, the texture. In the food industries, bulking agents are used to increase and stabilise the final volume of the feedstock and foodstuffs during the processing and storage. Frequently, the addition of sugars to the bulk contributes

significantly to the final volume. When moisturised with fat, it allows trapping air in its crystals, and when moisturised with flour, competes with gluten for water and dissipates the molecules of this protein. Both actions result in the enhanced lightness of the mass, improving its texture.

However, the need for low-calorie products has promoted the increased use of sweeteners, which do not help to give volume to the mass. Hence, the bulking agents are added to solid and semi-liquid foods, to increase the final products volume, without affecting the taste or contributing to the energetic final value of foodstuffs. Mannitol, methylcellulose, microcrystalline cellulose and polydextrose are some common bulking agents introduced into foods.

#### 2.1.6. Carbonation agents

The carbonation of beer, fruit juice, wine and soft drinks improves the tactual perception and provides slight acidity, a tangy taste and effervescence to these beverages. For this, carbon dioxide is added, according to the necessity of the industrial process and specific legislation. Carbonation is usually conducted at high pressure and low temperature to improve carbon dioxide solubility. Furthermore, the retention of substantial quantities of carbon dioxide in solutions at atmospheric pressure is attributed to surface adsorption by colloids and chemical ligations/interactions. The adequate setting of the carbonation parameters is important in the quantity of carbon dioxide liberated in industrial beverages and can be indicative of inferior quality (Damodaran & Parkin, 2017).

#### 2.1.7. Carriers

Some food additives can present low solubility or undesirable stability, interfering in its function and consequentially in the final quality and safety of foodstuffs. Compounds that aid in the dissolution, dilution, dispersion or physical modifications of a food additive, without modifying its technological properties are termed carriers, and their use facilitates the handling and application of food additives, such as flavouring and antifoaming agents. Some vegetable and mineral oils are commonly used as carriers in food processing (Msagati, 2012).

#### 2.1.8. Clarifying and appearance control agents

Turbidity and colour are important characteristics related to consumer acceptance of beverages, so the beverage industries employ certain additives to control these attributes. These compounds act to slow down the sedimentation of solids, change the viscosity and density, and disperse the flavouring by oil compounds, so they are referred to as appearance control additives (Damodaran & Parkin, 2017).

Clarifying agents are commonly added to improve the appearance of some beverages, such as fruit juice, beer and wine, by removing suspended particles or precipitates and preventing oxidative reactions, which are long-standing issues. Nonetheless, nonselective clarifiers can cause undesirable effects during the manipulation and can accidentally modify the content of the polyphenolic substances. Bentonite and montmorillonite clays are clarifying agents primarily used in wine due to their selectivity to preclude protein precipitation.

Polyamides and polyvinylpyrrolidone remove compounds that produce undesired flavour in beer. Some types of gelatine remove tannins, proanthocyanins and other polyphenols that compromise the quality of beverages (Belitz et al., 2009). Despite intensive use by the food industries, clarifying and appearance control agents are not considered among the chemical classes established by the *Codex Alimentarius* (FAO/WHO, 2016).

#### 2.1.9. Colours

The first characteristic of foodstuffs evaluated by consumers is the colour, which is related to the foodstuffs quality and directly influences the perception of flavour and sweetness. Moreover, undesirable colour modifications during the processing and storage of foodstuffs can be affected by many factors, such as the presence or absence of oxygen,

metals, light, oxidation reactions, pH and water activity. To overcome this issue, chemical compounds that impact colour, are intensively used by the food industries (Damodaran & Parkin, 2017).

The colours can be natural compounds, termed pigments, derived from the cells of animal and plant tissues. These colours do not cause a residual taste when added in sufficient concentration, and the health benefits of natural pigments, like carotenoids, carotenes, riboflavins, chlorophylls, betalains and other natural colours have been demonstrated (Msagati, 2012).

However, artificial colours are preferred by the food industries due to their stability, ease of handling during the processing of the foodstuff and control over the intensity of the colourant and thereby low cost. Table 1 presents the main colours used as food additives. Ponceau 4R, sunset yellow and amaranth, are solely used, and in some countries, their use is banned due to the promotion of diseases after accumulation in human organisms, similar to some other synthetic colours (Shibamoto & Bjeldanes, 2004).

#### 2.1.10. Colour retention agents

Appearance is the most important parameter evaluated by consumers before deciding whether to purchase the food. As mentioned in Section 2.1.9, the colour of the foodstuffs directly influences the perception of the freshness and food safety. Colour retention agents, like ferrous lactate, magnesium carbonate and magnesium hydroxide, play a crucial role in stabilising, preserving or intensifying the original food colour throughout the processing and storage (Msagati, 2012). Besides, the red appearance of meat products is improved by the addition of nitrates (FAO/WHO, 2017).

#### 2.1.11. Emulsifiers

The permanence of the sensorial, physicochemical and rheological properties of the foodstuffs is ensured by the use of emulsifiers, which provide a uniform dispersion between the dispersed phase (droplets) and continuous (bulk) phase, promoting a stable heterogeneous system, such as water and oil mixtures. Emulsifiers encourage stability, increase shelf life, control rancidity reactions, viscosity and texture, and the polymorphism and crystalline structure of fats, solubilise flavours and inhibit phase separation in foodstuffs, improving the acceptance of the industrialised products by consumers (Damodaran & Parkin, 2017).

The main emulsifiers used are the mono- and diacylglycerides and their derivatives, which correspond to around 75% of the emulsifiers worldwide. Many compounds (e.g., tartaric acid, succinic anhydride, ethylene oxide, citric acid) can be reacted with mono- and diglycerides of edible fatty acids to form new emulsifiers with different functions. Mono and diacetyl tartaric acid esters of mono- and diglycerides, for example, are used as dough conditioners in baked products, particularly, white bread, rolls and in flour mixes for convenience food. In chocolate production, emulsifiers are added to avoid defects in the appearance, such as phase separation that can occur (Belitz et al., 2009).

#### 2.1.12. Firming agents

The firmness and texture of processed products are industrially ensured through the use of firming agents that stabilise the cellular structure of fruits and vegetables. These rheological properties are maintained during the processing, independent of the temperature variation, using firming agents in sufficient concentration and inhibit the formation of a bitter flavour (Damodaran & Parkin, 2017).

Calcium salts, such as chloride, citrate, sulphate, lactate and phosphate, are commonly used to improve the effects of hardening before canning and freezing fruits and vegetables. Pectin and pectic acid remain in the cell wall during the heating of the fruits and vegetables due to the presence of firming agents added during the industrial process. This functionality can be improved by the combination of calcium and aluminium salts. In general, the efficiency of firming agents depends on the medium pH that is controlled by acetic and lactic acid additions (Belitz et al., 2009).

#### 2.1.13. Flavour enhancers

Sometimes it is necessary to intensify the flavour of foodstuffs to increase consumer acceptance. Improving the flavour perception at the molecule receptor level consequently promotes the alterations of the functions or neural systems feelings. For this purpose, flavour enhancers are added to foodstuffs during the processing steps (Belitz et al., 2009) to improve the palatability, characterised by sensorial parameters, such as complexity, fullness, body, food flavour, thickness and continuity. The use of flavour enhancers can also contribute to smell, improve the perception of softness, juicy and creaminess (Damodaran & Parkin, 2017).

Monosodium glutamate is the most known flavour enhancer, which promotes the sensory perception of the meat-like aroma notes in frozen, dehydrated or canned fish and meat products. However, in sensitive consumers, monosodium glutamate causes undesirable disorders, such as temporary drowsiness, headache, stomach ache and stiffening of the joints (Msagati, 2012).

The synthetic flavour enhancers, maltol and ethyl maltol, are often present in candy and foodstuffs produced from fruits, due to their ability to promote high softness and enhance sweetness. The nucleotides, such as 5'-inosine monophosphate and 5'-guanosine monophosphate are used as flavour enhancers in soups, sauces, canned meat and tomato juices, attributed to providing an increased viscosity of liquid foods and the sensations of freshness and naturalness (Belitz et al., 2009). Other flavour enhancers are shown in Table 1.

#### 2.1.14. Flour treatment and bleaching agents

Chemical compounds are frequently used by the flour industries to accelerate the maturation and ageing of flour, thereby improving the fermentation process and the appearance of the flour products. These substances are classified as flour treatment and bleaching agents, which promote the oxidative degradation of carotenoid pigments, by conversion to free radicals. The manipulation of the flour of pasta is facilitated through enzymatic inhibition and pH control, leading to an improvement in the flour colour and the texture of the flour products (Damodaran & Parkin, 2017).

Some flour treatments agents include ethyl maltol, disodium 5'-guanylate, disodium 5'-inosinate, guanylin acid, glutamic acid and others. Bleaching agents, like sulphites and benzoyl peroxides, improve the baking of the flour products without changing the colour of the foodstuffs (Msagati, 2012).

#### 2.1.15. Foaming agents

Some foodstuffs present foam in their physical structure to promote the aeration, improving the dispersion of the molecules and favouring the consumers' acceptance by further refining the sensorial characteristics. The food industries employ food additives, classified as foaming agents, in the stabilisation of the foams produced in the manufacturing steps, which are a prerequisite in the quality control of various foodstuffs (Green, Littlejohn, Hooley, & Cox, 2013).

Research has demonstrated that the chemical interactions between foaming agents and the components of the foodstuffs enrich the sensory quality of the products (Green et al., 2013). The interaction between the foam structure and the capacity of the foaming agent in the maintenance of this structure can be considerable in industrial processes. Hydrocolloids (gelatine and ovalbumin) are the most common foaming agents added during the processing of beers, ice creams, bread and emulsions. However, because these compounds originate from animal sources, they are rejected by some consumers, indicating the importance of developing alternatives with suitable quality and food security.

#### 2.1.16. Gelling agents

The gelling agents are a class of food additives used to promote gel formation, which is a viscoelastic semisolid that presents elastic solid and viscous liquid characteristics. The use of gelling agents produces a

three-dimensional physical structure, improving the homogeneity in the foodstuff. The gelling agents produce a gel with suitable stability, which can be a quality indicator, and act in the processing steps to enrich the viscosity of the final products (Belitz et al., 2009).

Hydrocolloids, often called gums, function as structuring/gelling agents via the involvement of specific inter-chain associations in conformationally ordered junction zones, which form the basis for the three-dimensional network characteristic of a gel. The physical arrangement of these junction zones is dependent on the pH, interest rheology, processing temperature, texture, quantity of gelling agents, interactions between the ingredients and the functional characteristics. The gums act as cloud agents, coating agents/film formers, clarifying agents, crystallisation and syneresis inhibitors, suspension stabilisers, binders, whipping agents, swelling agents, mould release agents, foam stabilisers, flocculating agents, fat mimetics, encapsulating agents, emulsion stabilisers, bulking agents, binders and bodying agents (Damodaran & Parkin, 2017).

The aligins, carboxymethyl-cellulose, carrageenan, pectin, alginate, xanthan and others, shown in Table 1, are the most common gelling agents used by the food industries. The types and the quantity used are defined by the specific legislation, according to each country (Msagati, 2012).

#### 2.1.17. Glazing agents

The appearance of some foodstuffs can become more attractive, when they look brighter, such as fruit and vegetable candies. Providing a brighter exterior and protection to the external surface of foodstuffs can be achieved by the addition of glazing agents during the manufacturing process. The protective outer coating provided by some glazing agents, such as the waxes in apple peel, can also increase the shelf life of some fruits and vegetables (FAO/WHO, 2017).

In industrial processes, the most common glazing agents used are beeswax, candelilla wax, carnauba wax, castor oil and polyethylene glycol, among others shown in Table 1.

#### 2.1.18. Humectants

During the transport and storage, some foodstuffs can lose moisture, decreasing the shelf life and damaging the appearance of the product. In general, the loss of moisture affects the final texture and, consequently, sensorial perception, attributed to the unpleasant dryness or hardening of the food experienced by the consumers. To avoid this problem, humectants, i.e., substances that protect foods from the moisture loss, may be used in the formulation of foods susceptible to dryness. Glycerol, mannitol, polydextrose and propylene glycol are humectants employed in the fabrication of sweets, bread, cake and biscuit fillings, chocolates and other products (Msagati, 2012).

#### 2.1.19. Preservatives/antimicrobial agents

The prevention of the spoilage and assurance of the safety of food can be obtained by use of compounds that act as preservatives of foodstuffs, by presenting antimicrobial properties, preventing the degradation by enzymatic and non-enzymatic reactions. Its realisation depends on some conditions, such as the pH and solubility of the food. The use of preservatives/antimicrobial agents increases the shelf life of the foodstuffs, but at high concentrations, these compounds can promote an unpleasant taste, intense odour, change in viscosity and colour retention, and decrease in the solubility (Msagati, 2012).

The food industries employ preservatives/antimicrobial agents, which can be used to inhibit non-enzymatic browning and enzyme-catalysed reactions, and the reversible cleavage of proteins (sulphites and sulphur dioxide). In the meat industries, the sodium and potassium salts of nitrite and nitrate are often used to preserve cured meat and sausages. In the production of bread and baked goods, the acetates of sodium, potassium and calcium, and propionic acid were incorporated to prevent the growth of moulds, thereby increasing the shelf life of these foodstuffs. Other compounds used for this purpose are benzoate,

sorbate, epoxides, glycerol esters, and diethyl pyrocarbonate (Belitz et al., 2009).

Some antibiotics are used as food preservations, but their use must be adequately evaluated, as the consumption of food treated with antibiotics may cause sensitisation of the consumer to the antibiotics used and may result in the emergence of antibiotic-resistant microorganisms. In the cheese and yoghurt industries, the inhibition of undesirable bacteria, yeasts and fungi is realised by the use of preservatives, such as benzoates, sorbates, natamycin (an antibiotic) and *Lactobacillus*, which maintain the quality of products and improve the nutritional quality, respectively (Damodaran & Parkin, 2017).

#### 2.1.20. Propellants

Some foodstuffs are physically presented as sprays, foams and liquids. The stabilisation of these forms is a huge challenge in the food industries. Pressurised aerosols, called propellants, promote the expansion of the foodstuffs and are in intimate contact with the food, and therefore, are considered as food additives. Propellants are classified according to their physical state (liquids and gaseous compounds), and their use depends on the food physicochemical property's needs (Damodaran & Parkin, 2017).

Gaseous propellants are non-toxic, do not promote undesirable flavour and colour, are non-flammable and present low cost. The most common gaseous propellants are nitrous oxide and carbon dioxide, employed in systems where foams and sprays are desired, due to their water solubility and suitable expansion in closed systems. Carbon dioxide is used in products where the tanginess and tartness are necessary, such as in cheese. Nitrogen is used in ketchup, edible oils and syrups, in which the foaming can be avoided (Belitz et al., 2009).

The main liquid propellants used in vegetable oils and emulsified products are octafluorocyclobutane and chloropentafluoroethane. These compounds present low flammability, are non-toxic in low concentrations and do not interfere with the flavours of the foodstuffs. Other chlorofluorocarbon compounds are prohibited, as they are environmentally toxic, promoting degradation of the ozone layer in the atmosphere (Msagati, 2012).

#### 2.1.21. Raising agents

Raising agents or chemical leavening systems are compounds added to foodstuffs to promote the expansion of the dough or batter, improving the texture, softness and flavour. The use of raising agents fosters the release of carbon dioxide and the expansion of pasta, which is dependent on the concentration and temperature of paste, that interfere directly in the characteristic of the foodstuffs based on baking mixes, household, self-rising flours, commercial baking powder and dough products (Damodaran & Parkin, 2017).

The more common raising agents contain bicarbonate salt and carbonate that are soluble in water, promoting complete ionisation of carbonate and release of carbon dioxide. The dough or batter are complex systems, containing proteins and other natural compounds that can participate in the chemical reactions of the raising agents, avoiding the appearance of undesirable flavour in the foodstuffs and assisting in the end pH stabilisation. Other salt acids used as raising agents are potassium acid tartrate, sodium aluminium sulphate,  $\delta$ -gluconolactone and acid pyrophosphate, which produce gas due to specific enzyme actions (Msagati, 2012).

#### 2.1.22. Sequestrates/chelating agents

Sequestrants or chelating agents are chemical compounds used as stabilisers of foodstuffs, through complexation of metals ions that catalyse hydrolytic reactions and degradations process. Metals interfere in foodstuffs, modifying the flavour, smell and colours and, consequently, promote alterations in the final product stability. Some natural compounds present in the feedstocks act as sequestrants or chelating agents, such as chlorophyll that complexes with magnesium; ascorbic acid that complexes iron to improve its ingestion; some enzymes that complex

copper and zinc; and some proteins that complex iron, among others (Belitz et al., 2009).

In industrial processes, the sequestrant or chelating agents employed are chemical compounds that present the functional groups  $-NR_2$ ,  $-S-$ ,  $-OH$ ,  $-SH$ ,  $-O-$ ,  $C=O$ ,  $-COOH$  and  $H_2PO_3^-$ , which have an unshared pair of electrons that can react with metals ions. The complexation reactions depend on the pH of the medium. Polyphosphates and ethylene diamine tetra-acetic (EDTA) salts can be used in canned seafood to prevent the formation of glassy crystals, and in vegetables and fruits to inhibit the discolouration reactions. Some acidulate also act as sequestrants or chelating agents, such as citric acid and phosphoric acid, used in the soft drinks industries (Damodaran & Parkin, 2017). The EDTA salts are the most used sequestrants employed by the food industries, but their use requires rigorous control due to a capacity to complex with metals naturally present in humans. The inadequate ingestion of certain minerals can promote serious diseases (Msagati, 2012).

#### 2.1.23. Stabilisers

The food industries frequently use compounds to avoid phase separations, ensuring the homogeneity of foodstuffs. These compounds are classified as stabilisers, and their use is fundamental to ensuring a desirable sensorial attribute, particularly, in the stabilisation of emulsions and suspensions, to improve the texture by inhibiting the formation of crystals and increasing the viscosity, thereby enhancing the appearance of foodstuffs (Damodaran & Parkin, 2017).

Stabilisers are also utilised as thickener agents, gelling agents, suspending agents and glazing agents, as well as to control the fluidity and retention of water in diverse products. The properties of stabilisers are influenced by the pH, temperature, concentration, molecular weight and ionic forces. These foods additives are used in broths, desserts, sweets, preserves, ice creams, pasta, dairy products, processed foods, bakery, soups, biscuits, juices and chocolates. Examples of some common food stabilisers are alginate, carrageenan, casein, carboxymethylcellulose sodium salt, xanthan, guar and locust bean gum (Belitz et al., 2009).

#### 2.1.24. Sweeteners

Sweeteners are chemical compounds used to intensify the sweetness and/or the perception of the sweet taste, directly influencing the acceptance of foodstuffs. Artificial or non-nutritive sweeteners are often used as a sugar alternative. These sweeteners are low in calories. Such compounds must demonstrate safety at the levels used in the food and have a low residual taste. Nutritive sweeteners (e.g., fructose, isomalt, sugar alcohols, maltodextrin) are based on different types of carbohydrates and have less energy than sugar, but they are not calorie-free. Sweeteners are chemically divided into sulphonamides, peptides, chlorosaccharides, low-calorie sweeteners and polyols (Damodaran & Parkin, 2017).

The sulphonamide sweeteners present a sulphamic acid group and comprise potassium acesulfame, cyclamates and saccharin. These compounds are up to 700 times sweeter than sucrose but cause a bitter and residual metal taste. Research about the toxicities of the sulphonamides are controversial, but an increased incidence of diseases attributed to the ingestion of these substances has not been proven (Shibamoto & Bjeldanes, 2004).

The peptide sweeteners present suitable sweetness at low concentrations and, consequently, their use promotes insignificant ingestion of calories. These compounds have a sweetness intensity of 200–13,000 times that of sucrose, without residual taste. Despite this, their use can promote alteration in the flavour of foodstuffs. Moreover, for some consumers, the use of these sweeteners can provoke health problems, such as allergies, heart diseases and phenylketonuria (Belitz et al., 2009).

The chlorosaccharide sweeteners present a more intense sweetness potency (600-fold) than sucrose (sugar) and are produced by selective

chlorination of the saccharide. These compounds have high crystallinity, no bitterness, and good solubility and stability at various temperatures (Msagati, 2012). The polyols are a chemical class that beyond sweetening, promote control of the texture, crystallisation and viscosity, humidity retention and diminution in the water activity, improving the softness and water rehydration properties of foodstuffs (Damodaran & Parkin, 2017).

#### 2.1.25. Thickeners

In choosing foodstuffs, the consumer evaluates the texture and viscosity, which can influence the perception of the food quality either positively or negatively. In some foods, like ice cream, chocolate milk, cream cheese, mayonnaise and confectionery product coverings, high viscosity is desirable. Therefore, to obtain the necessary viscosity, the food industries add thickeners in the food processing steps. These added compounds are water-soluble and hydrophilic, being used to disperse, stabilise or prevent sedimentation of substances in suspensions. Gums, starches, pectins and others (Table 1) are most commonly used for this purpose.

### 3. Analysis of food additives

The use of food additives requires a rigorous food safety policy since some compounds can pose a risk to the consumers' health. Some researchers have shown that the cumulative consumption of food additives can provoke allergies, diabetes, obesity and metabolic disorders (Shibamoto & Bjeldanes, 2004). As a result, each country has specific legislation, encouraging a suitable quality control in industrialised foodstuffs, with the identification and quantification of food additives used or produced during the industrial process. Illegal food additives, used in some industries to mask the degradation process or inadequate manipulation procedures, also need careful monitoring.

Analytical techniques are used to identify and quantify different food additives in feedstocks, independent ingredients, the processed products and end-products, and in all intermediate compounds formed during the manufacturing process. Thus, it is necessary to implement techniques that provide reliability, selectivity, sensitivity, quickness, operational security and are environmentally friendly and, mainly, low cost, to use in quality control and evaluation of food safety (Wroldstad et al., 2005).

Regardless of the instrumental techniques (spectroscopic, chromatographic and electroanalytical) employed, the food additives analysis requires the adequate evaluation of the sampling and pre-treatment steps and the interpretation of the results, which are responsible for success in chemical analysis (Mitra, 2004). The strict sampling procedure is necessary to guarantee the representativeness of the food sample, by reflecting all its constituents and so obtaining reliability in the analytical results. In an ideal sampling procedure, the sample should be identical considering intrinsic properties from the raw materials, independent ingredients, the processed products or end-products analysis (Nollet & Toldrá, 2017).

To ensure the representativeness of the samples and, consequently, the reliability of the analytical results, the sampling protocol is optimised by statistic techniques. The sampling protocol depends on the size of the sample, variability of the sample components, cost of the analysis of each sample and, sometimes, depends on the specific legislation regulating the use and analysis of the food additives, indicating the procedures and minimum amount of the samples necessary in the chemical analysis (Nielsen, 2014).

The presence of fats, oils, lipids, proteins, carbohydrates, polysaccharide, salts, surfactants, pigments, emulsions and turbidity can interfere in the analysis of food additives. Hence, pre-treatment of the food samples is conducted before the chemical analysis, to remove as many interfering compounds as possible. The adequate choice of the sample preparation method is based on the nature of the food sample and in the analysis to be performed. Mixing, homogenisation, dilution, centrifugation, distillation, simple solvent extraction, supercritical fluid

extraction, pressurised-fluid extraction, microwave-assisted extraction, Soxhlet extraction and/or derivation, are some procedures effectuated as pre-treatments of food samples. These pre-treatments need to be rapid, simple, inexpensive and environmentally friendly (Wroldstad et al., 2005).

The use of microwave-assisted extraction as a pre-treatment method in high-performance liquid chromatography (HPLC) and near-infrared (NIR) analysis have been demonstrated enhanced recovery of food additives, limiting extraction time and solvent waste (Nescatelli et al., 2017). The employment of magnetic molecularly imprinted polymers for food additive analysis promote a less time-consuming pre-concentration procedure comparing to classical methods, with fast adsorption kinetics and selective adsorption capacity (Xu, Niu, Chen, Zhao, & Chen, 2017). Ultrasound-assisted extraction has been shown as a suitable pre-treatment method in the food additives analysis, due to the enhancement of the analytical responses attributed to cavitation and thermal effects of ultrasonication.

The analytical data in food analysis can be validated using statistical methods, ensuring reliability and analytical quality and adequacy to specify legislation. The linearity, accuracy, precision, sensitivity, range concentration, limit of detection, limit of quantitation and robustness are analytical parameters evaluated to demonstrate the analytical method employed is capable of identifying and quantifying the food additive, with reliability in complex samples and over a suitable range of concentrations (Wroldstad et al., 2005).

There are many analytical techniques available for the analysis of the food additives. The ultimate choice depends on the nature of the samples (solid, liquid or gas), expected level of the additive, the analyst's experience, the equipment and reagents available, the time of analysis, the number of samples and, mainly, the levels of interfering substances present in the samples.

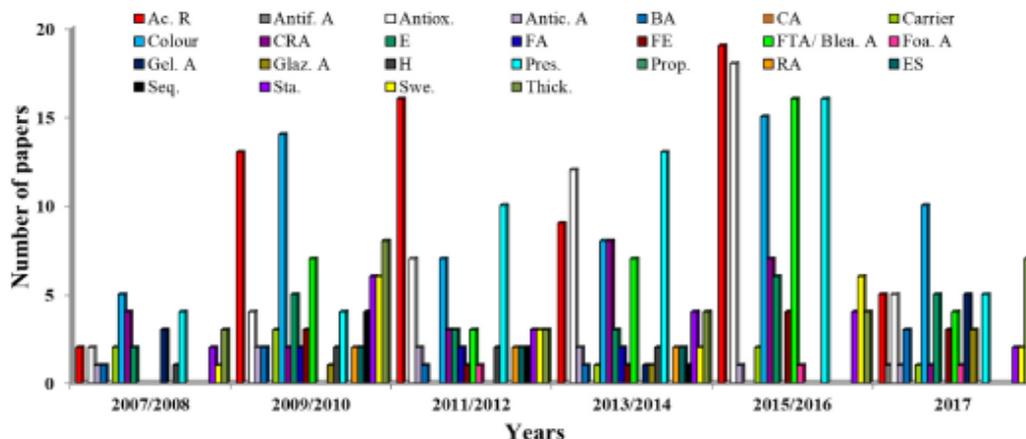
Figs. 2–4 show the research realised during the last 10 years, to indicate the applicability of the spectroscopic, chromatographic and electroanalytical techniques, respectively, for the analysis of the 25 functional classes of food additives. Most of the published papers are related to the analysis of food additives that promote toxicities to the consumer and/or cause undesirable flavour in the foodstuffs, which are acidity regulators, colours, antioxidants and preservatives.

Each technique presented has a set of characteristics, allowing to identify and quantify the additive of interest, with advantages and disadvantages typical of the methodology used. The main analytical methods used in the analysis of the food additives in foodstuffs are classified in spectroscopic (Section 3.1), chromatographic (Section 3.2) and electroanalytical techniques (Section 3.3).

#### 3.1. Spectroscopy techniques

The spectroscopy techniques are based on the interaction of electromagnetic radiation with the matter. The energy from heat, electric discharge or light promote the momentary excitation of the electrons to a state of greater energy (excited state), and when the electrons return to the initial energy status (fundamental states), energy is released, previously absorbed, in the light form. If the light intensity measured is higher than the added light on the sample occurs higher light emission (emission spectroscopy) and if the light intensity measured is lower than the added light on the sample occurs absorption and the measured intensity of light is lower (absorption spectroscopy) (Skooog, West, Holler, & Crouch, 2014).

Chemical structures of food additives present functional groups, allowing the absorption and/or emission of electromagnetic radiation at a known wavelength, which can be employed to quantify food additives in feedstocks and foodstuffs. The main methods employed by the food industries are ultraviolet/visible radiation (UV/Vis) spectroscopy and infrared spectroscopy (IR), which are used in determining the chemical composition, textural parameters and quality-related parameters (Wroldstad et al., 2005).



Ac. R = Acidity Regulator; Antic. A = Anticaking Agent; Antif. A = Antifoaming Agent; Antiox. = Antioxidant; BA = Bulking Agent; CA = Carbonating Agent; CRA = Colour Retention Agent; E = Emulsifier; ES = Emulsifying Salt; FA = Firming Agent; FE = Flavour enhancer; Foa. A = Foaming Agent; Gel. A = Gelling Agent; Gla. A = Glazing Agent; H = Humectant; Pres. = Preservative; Prop. = Propellant; Seq. = Sequestrant; Swe. = Sweetener; Thick. = Thickeners;

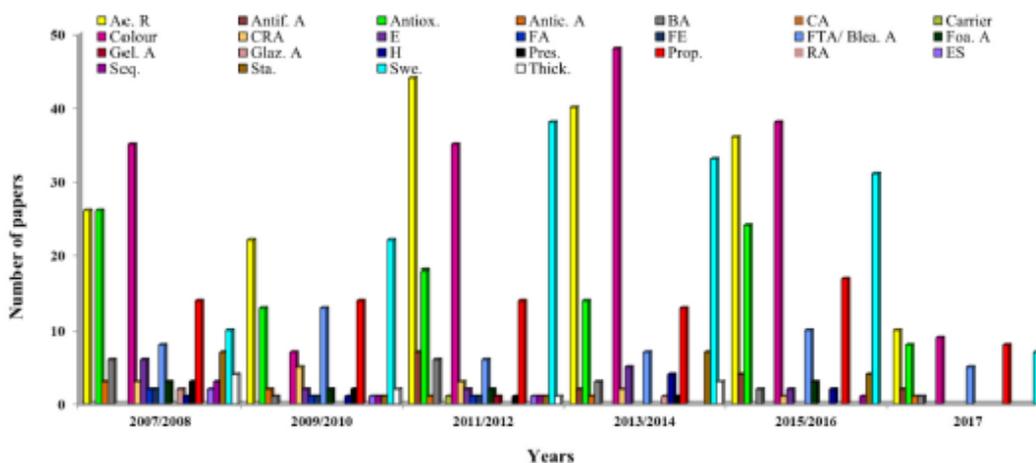
Fig. 2. Researches related to the analytical determinations of food additives in foodstuffs using spectroscopic methods, from 2007 to 2017 years.

The UV/Vis technique (from 190 to 800 nm wavelength) measures the fraction of radiation transmitted through the sample. Its instrumentation requires a source of electromagnetic radiation, a means of selecting the wavelength at which the transmittance is measured and a detector for measuring the transmittance. The transmittance is related to the absorbance and the concentration of the absorbing species, by Beer's law (Christian, Dasgupta, & Schug, 2014).

Use of UV/Vis in quality control is adequate in the determination of different food additives, attributed to its efficiency, low cost, non-destructive and environmentally friendly approach, and operational

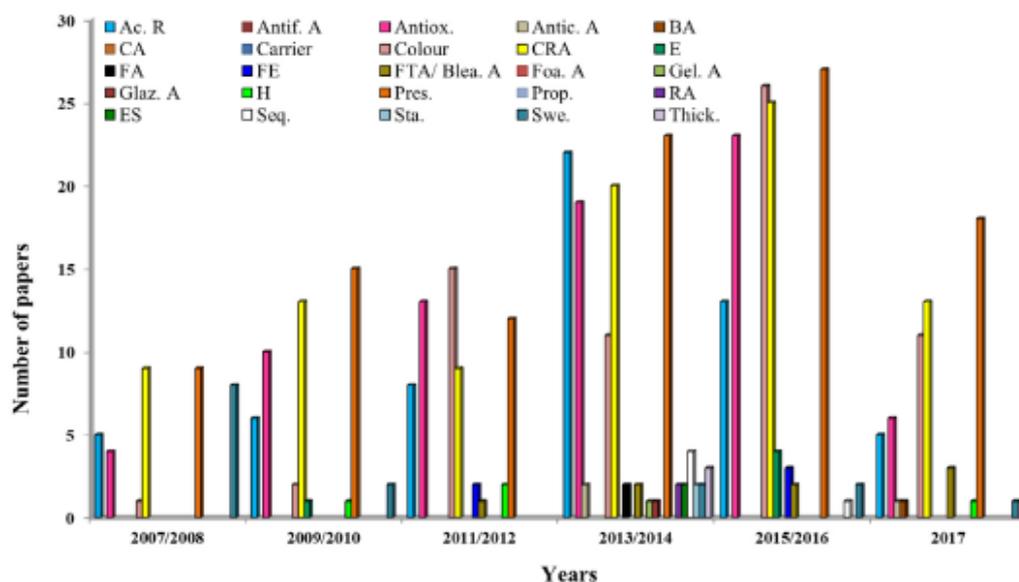
simplicity, and is considered suitable for online measurement. However, in foodstuffs analysis, the composition of the samples promotes an undesirable selectivity, due to the presence of other chemical compounds that absorb electromagnetic radiation. To overcome this issue, it is necessary to first treat the sample, for further analytical efficiency and selectivity (Skoog et al., 2014). The use of artificial neural network methods is used to model multicomponent absorbance data and improve the selectivity of food analysis by UV/Vis techniques (Esteki, Shahsavari, & Simal-Gandara, 2018; Santos & Schug, 2017).

In IR spectroscopy, the absorption of light by chemical compounds



Ac. R = Acidity Regulator; Antic. A = Anticaking Agent; Antif. A = Antifoaming Agent; Antiox. = Antioxidant; BA = Bulking Agent; CA = Carbonating Agent; CRA = Colour Retention Agent; E = Emulsifier; ES = Emulsifying Salt; FA = Firming Agent; FE = Flavour enhancer; Foa. A = Foaming Agent; Gel. A = Gelling Agent; Gla. A = Glazing Agent; H = Humectant; Pres. = Preservative; Prop. = Propellant; Seq. = Sequestrant; Swe. = Sweetener; Thick. = Thickeners;

Fig. 3. Researches related to the analytical determinations of food additives in foodstuffs using chromatographic methods, from 2007 to 2017 years.



Ac. R = Acidity Regulator; Antic. A = Anticaking Agent; Antif. A = Antifoaming Agent; Antiox. = Antioxidant; BA = Bulking Agent; CA = Carbonating Agent; CRA = Colour Retention Agent; E = Emulsifier; ES = Emulsifying Salt; FA = Firming Agent; FE = Flavour enhancer; Foa. A = Foaming Agent; Gel. A = Gelling Agent; Gla. A = Glazing Agent; H = Humectant; Pres. = Preservative; Prop. = Propellant; Seq. = Sequestrant; Swe. = Sweetener; Thick. = Thichener;

Fig. 4. Researches related to the analytical determinations of food additives in foodstuffs using electroanalytical methods, from 2007 to 2017 years.

promotes a change in vibrational energy levels, resulting in the absorption of IR radiation by different functional groups, at different frequencies of radiation. The absorption of radiation in the near-IR, from 0.8 to 2.5  $\mu\text{m}$  wavelength, is useful for qualitative analysis, such as identifying specific functional groups present in substances while mid-IR, from 2.5 to 15  $\mu\text{m}$  wavelength, is used in quantitative analysis. IR spectroscopy techniques are widely used to identify functional groups in solid, liquids and gaseous samples.

NIR uses a radiation source that provides intense radiation in the 0.8–1.75  $\mu\text{m}$  range and a detector roughly 100 times more sensitive than other IR detectors. Hence, this technique is used most extensively for quantitative applications, either using transmission or diffuse reflection measurements that can be taken directly from solid foodstuffs. For this reason, NIR finds use in the analysis of feedstocks and final products, such as biscuits, muesli bars, ready-to-eat breakfast products, processed meat products, butter and margarine.

The NIR instrumentation uses multivariate statistical techniques and calibration steps to measure the quantities of various food additives in a variety of foodstuffs, based on the amount of IR radiation absorbed at specific wavelengths. Besides, the method performs rapid quantitative analysis, does not require hazardous reagents and no chemical waste is generated. However, the high initial cost of the instrumentation and the necessity of the specific calibrations for each product measured are the disadvantages of NIR spectroscopy in the food industries.

In Fourier transform infrared (FTIR) spectroscopy the instrumentation uses an interferometer. The radiation is not dispersed, but rather all wavelengths arrive at the detector simultaneously. The results are converted to give a typical IR spectrum of the absorbance vs frequency of the radiation, using the Fourier transformations as the mathematical procedure to improve the quality and interpretation of the analytical results, facilitating the use of IR in food quality control. This technique

provides a rapid and accurate data acquisition and is a non-destructive procedure. It also allows data analysis by a variety of software algorithms that have dramatically increased its practical use in the food industries (Babushkin, Spiridonov, & Kozhukhar, 2016). In addition, FTIR spectroscopy permits determining the concentration of each food constituent from the absorbance values at a selected wavelength, improving the selectivity in the food analysis. However, like NIR spectroscopy, the high initial cost of the instrumentation is the main drawback of FTIR in the food industries (Amir et al., 2013; Van de Voort, 1992).

Raman spectroscopy is a technique based on the scattering of light, where the frequency of a small fraction of scattered radiation is different from the frequency of monochromatic incident radiation in the sample. The Raman spectra are formed from the inelastic collision between incident monochromatic radiation and molecules of the sample and can be used for qualitative and quantitative purposes. The lasers used in Raman spectroscopy emit radiation in the near-IR wavelength, and the intensity of the scattered light is measured at different frequencies from the incident light. Raman spectroscopy is applicable to liquids and solid foodstuffs. The Raman analysis is quick and cheap, due to a simple preparation of the samples; however, the weak Raman scattering promotes a low analytical sensitivity (Bumrah & Sharma, 2016; Nielsen, 2014).

Fluorescence spectroscopy is a technique based on the emission of the electromagnetic radiation by chemical compounds when a molecule in the lowest vibrational energy level of an excited electronic state returns to a lower energy electronic state, by emitting a photon after the absorption of the light of a specific wavelength (Sádecká & Tóthová, 2007). The fluorescence spectra are formed by respective wavelengths for excitation and emission and will depend on the chemical structure of the target compound. Only chemical compounds that present rings and rigid chemical structures will emit electromagnetic radiation with

considerable intensity because these structures do not allow the release of the energy absorbed by paths other than the emission of light (Christian et al., 2014).

Fluorescence instrumentation is like UV/Vis spectroscopy, but the analytical results are 1–3 orders of magnitude more sensitive than UV/Vis. However, fluorescence is only used for rigid chemical structures analysis, and few compounds in food present these structures, decreasing the applicability of this technique in the food industries (Albani, 2012). The high selectivity of fluorescence is used as an accurate tool to monitor the molecular changes that occur during food handling, processing or storing of foodstuffs containing sweeteners, colours and emulsifiers (Christian et al., 2014).

Table 2 provides recent research that developed and applied analytical procedures using spectroscopy techniques in the analysis of various classes of food additives in a range of feedstocks and/or foodstuffs.

### 3.2. Chromatographic techniques

Chromatographic techniques are a set of analytical techniques employed to separate, identify and quantify inorganic and organic compounds, based on the partitioning or distribution of a sample between a mobile and a stationary phase. The separation occurs due to a series of equilibria between the species of interest and the mobile (gas, or liquid or supercritical fluid) and stationary phase (solid or liquid adsorbed onto a solid support).

Chromatographic techniques are classified according to the physicochemical principles involved in the separation. In adsorption chromatography, the stationary phase is a solid on which the sample components are adsorbed, whereas, in partition chromatography, the stationary phase is a liquid supported on a solid. Ion exchange chromatography employs an ion exchange support as the stationary phase. Exclusion chromatography separates the solvated molecules by their ability to penetrate porous pockets and passages in the stationary phase, associated with their size. These separation mechanisms operate in the two principal types of chromatography used in food additive analysis, gas chromatography (GC) and liquid chromatography (LC) (Jonsson, 1987).

GC is a versatile technique that separates and identifies complex mixtures of volatile compounds and thermally stable organic compounds, using an inert gas as the mobile phase and either an immobilised liquid or a solid packed in a closed tube, as the stationary phase (Nielsen, 2014). The GC instrumentation consists of an inert gas supply, pressure and flow control regulators, injection port of the samples, column, detector, and a data recording and processing system.

Preliminarily, the sample is converted to the vapour state and injected into a column with a controlled temperature gradient to separate the molecules according to several properties, including boiling point, molecular size and polarity. The sample emerges from the column at a constant flow rate and arrives at the detector where the specific response is dependent upon the analyte.

A wide variety of detectors, such as thermal conductivity, flame ionisation, electron capture, flame photometric, photoionisation, electrolytic conductivity and mass spectrometry (MS) are commercially available. The appropriate choice will depend on the nature of the sample to be analysed, and their use can provide either sensitivity or selectivity in the analysis. The detector signal or chromatographic peaks are acquired and displayed by a data system as a function of time. The area under the peak is proportional to the concentration, and so the amount of substance can be quantitatively determined by the construction of a calibration curve prepared in the same manner (Wroldstad et al., 2005). Recently, a vacuum UV spectrophotometer was developed to use as a GC detector, which measures the absorption of gas-phase chemical species in the range of 120–240 nm, where all chemical compounds present unique absorption spectra, permitting a facile deconvolution of co-eluting food additives (Santos & Schug, 2017).

The use of GC requires that the sample be volatile and stable at the operational temperatures, typically from 50 to 300 °C. Derivatisations steps to convert compounds to volatile or thermally stable compounds can be realised, but derivatisation is not always possible and/or efficient. Besides, GC analysis of food additive from foodstuffs necessitates rigorous preparation steps, which generally involve preparative chromatography, such as solid-phase extraction or liquid-liquid extraction, headspace analysis and distillation. GC instrumentation has an excessive cost in initial acquisition, maintenance and in the utilisation of the equipment. Furthermore, the analyst must have high expertise and technical knowledge of the technique, restricting its use in quality control to small industries.

Despite the advantages of GC, approximately 80% of known compounds are not sufficiently volatile or stable to be separated by GC. Instead, the use of LC, especially HPLC, is highly adequate, where the mobile phase is a liquid, and the stationary phase is a solid, which can be adequately chosen to improve the separation and identification of different chemical classes of organic and inorganic compounds, according to the polarity of the target compounds.

The LC analyses are based on the adequate choice of a mobile phase, composed by organic solvents and/or buffer solutions, which promote a suitable separation of the target compounds based on their differential affinity between a solid stationary phase and a liquid mobile phase. Classical LC operates at near atmospheric pressure by utilising a peristaltic pump or gravity flow to maintain a constant flow of the mobile phase liquid. HPLC originated from classic LC through the use of pumps and specific detectors that promote improvements in the separation, identification and quantification of organic compounds (Wroldstad et al., 2005).

The basic HPLC instrumentation is composed of a pump, injector, column, detector and data system. The mobile phase, called the eluent, use highly pure organic solvents and buffer solutions, which are pumped at a constant flux through the column, promoting the separation of the components by differences in the affinities between the mobile and stationary phase. The pumps allow the isocratic elution arrangements (constant proportion of solvents during the separation) or gradient elution (variation in the ration organic solvents and buffer solutions during the separation) (Christian et al., 2014).

Pumping systems capable of running a gradient containing up to four components are largely employed in quality control laboratories. The injectors are used to place the sample in the flowing mobile phase, for introduction onto the column. The most common are valve-type injectors and autosamplers, which are employed to inject large numbers of samples, such as in food control analysis.

The type of column employed defines the type of chromatography. Normal phase chromatography utilises a polar stationary phase and relatively non-polar-to-intermediate polarity solvents (hexane and tetrahydrofuran) as the mobile phase. Reverse phase chromatography uses polar organic solvents, such as acetonitrile and methanol joint buffer solvents. Ion exchange chromatography uses ion exchange resins that are composed of particles carrying fixed positive or negative charges. In anion exchange, the stationary phase contains many positively-charged groups (typically quaternary ammonium ions). Negatively-charged species are attracted to the resin and travel more slowly than the column than positively-charged species. Similarly, positively-charged species are attracted to cation exchange resin's negatively-charged groups (sulfonate groups are often the side-chains in cation exchange resins). Ion chromatography uses microparticulate ion exchangers, and the identification and quantification are achieved by conductometric detection. In size exclusion chromatography, the molecules are separated based on their size (Christian et al., 2014).

The HPLC columns are composed by high-purity silica particles with diameters less than 2 µm, to create high back pressure and present minor loss of efficiency at high flow rates, permitting rapid separations. The silanol groups present in silica particles provide polar interaction sites, and these groups can be functionalised, modifying the polarity of

**Table 2**  
 Spectroscopy, chromatography and electroanalytical techniques used in food additives analysis.

Classes	Food additives	Detector	Samples	LD	Ref.
Flavour enhancer	Maltol	FAAS	To-mato soup, dark chocolate, plain biscuits, fruit cake, pudding, milk wafers, lemon and grape apple juice, strawberry, chocolate and banana milk, fruit yogurt and milk powder	1.24 µg L <sup>-1</sup>	(Ahumay, Ghifan, & Orhan, 2017)
Flour treatment agent	Axodiphenolamide	NIR	Flour	3.2 mg kg <sup>-1</sup>	(Che et al., 2017)
Acidity regulator and colour reaction agent	Magnesium hydroxide	AAS	Almond gum and water	0.05 µg L <sup>-1</sup>	(Shokrollahi, Hemmatidoust, & Zargampour, 2016)
Colours	Garcinoids	Raman and UV/Vis	Carrot	NR	(Lawrenz et al., 2016)
Flavour enhancer	Glutamic acid	IR	Cereals	NR	(X. Zhang, Lu, Luo, & Zhang, 2017)
Sweetener	Acnesulfame potassium	NMR	Chewing gum, red bean paste, candy, jelly, biscuit, ice cream, jam, salad dressing, vegetable pickled in soy bean sauce, sherbet, soft drink and beverage containing <i>Lactobacillus</i>	0.13 g kg <sup>-1</sup>	(Ohtsuki, Sato, Abe, Sugimoto, & Akiyama, 2015)
Colours	Curcumin	FLU	Supplements and spices	0.1 mg L <sup>-1</sup>	(Petrova et al., 2016)
Glazing agent	Mineral oil	FTIR	Oil	NR	(Polanski, Proser, Vlk, Moravcova, & Pihara, 2017)
Sweetener	Aspartame, cyclamate, saccharin	FTIR and Raman	Tabletop sweeteners	NR	(Daute et al., 2017)
Colours	Alura red	FLU	Candies and beverages	0.002 µg mL <sup>-1</sup>	(Bakhet & Zhu, 2017)
Colour	Ponceau 4R	FLU	Beverages	15 µg mL <sup>-1</sup>	(J. Zhang et al., 2017)
Colour	Tartrazine, Sunset Yellow, Ponceau 4R and Fast Green	DRS	Barberry taste, orange juice	0.2–0.6 mg L <sup>-1</sup>	(Tikhomirova, Rumazanova, & Apyari, 2017)
Colour	Sunset yellow	FLU	Soft drinks	79.6 nmol L <sup>-1</sup>	(Yuan et al., 2016)
Colour	Riboflavin	Cl. and FLU	Tablet	0.63 nM	(H. Wang et al., 2017)
Colour	β-carotene	Raman	Oil-in-water emulsions	0.1–0.3 g kg <sup>-1</sup>	(Wan Mohamad, Budlow, Augustin, & McNaughton, 2017)
Acidity regulator and antioxidant	Ascorbic acid	FLU	Hawthorn and jujube	39 nmol L <sup>-1</sup>	(Xu, et al., 2017)
Colour	Curcumin	UV/Vis	Ginger powder, curry powder and ginger	1.31 µg mL <sup>-1</sup>	(Liu et al., 2016)
Bleaching agent and flour treatment agent	Benzoyl peroxide	FTIR	Wheat flour	NR	(X.X. Guo et al., 2016)
Emulsifier, gelling agent, glazing agent, and foaming agent, bulking agent, foaming agent, stabilizer and thickener	Alginate and carrageenans	NMR	Seaweeds	NR	(Youssef et al., 2017)
Emulsifier, stabilizer and thickener	Guar gum	Raman	Arabic, gellan, karaya, tamarind, xanthan, locust bean, rosin and ghatti gums	NR	(Srinivasa, Wolfgang, & Rodriguez, 2016)
Preservative and colour retention agent	Nitrites	Cl.	Seawater	0.45 nM	(Foreman, Segura-Nogueras, & Kari, 2016)
Sweetener	Sodium saccharin	Raman	Soft drinks	0.3 mg L <sup>-1</sup>	(Han et al., 2017)
Preservative and colour retention agent	Nitrite	UV/Vis	Food	3 µg L <sup>-1</sup>	(Nsofor, Tan, Heng, Chong, & Tajuddin, 2016)
Thickener, bulking agent, emulsifier, gelling agent and stabilizer	Sodium alginate	FLU, NMR and IR	Seaweed	NR	(Ferrah & Belkiza, 2014)
Preservative and colour retention agent	Nitrite	UV/Vis	Vegetables, fruits, grains, legumes, dairy products, meat	0.10 mg/100 g <sup>-1</sup>	(Bahadron et al., 2016)
Stabilizer, carrier, emulsifier and thickener	Dextrin	FTIR and NIR	Sorbitol molarities	NR	(T. Guo et al., 2016)
Chromatography					
Classes	Food additives	Column/ detector	Samples	LD	Ref.
Preservative and colour retention agent	Nitrite	GC-MS	Raw lettuce, spinach, carrot, kale, arugula and endive	0.2 µg g <sup>-1</sup>	(Cimpanella, Onor, & Pagliaro, 2017)

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Table 2 (continued)

Classes	Food additives	Detector	Sample	LD	Ref.
Glazing agent	Mineral oil	HPLC-CC-RID	NR	NR	(Blodermann, Muzoz, & Grob, 2017)
Anticaking agent, humectant, bulking agent, stabilizer, sweetener and sweetener	Mumitol	HPLC-UV/VIS	Fermented rice	52.1 µg mL <sup>-1</sup>	(Chiang, Liang, Wang, & Liang, 2017)
Acidity regulator	Lactic acid, fumaric acid and malic acid	GC-MS	Grape wine	0.07 ng mL <sup>-1</sup>	(Tang & Duan, 2017)
Flavour enhancer, sweetener, acidity regulator, preservative	Aspartame, cyclamate, saccharine, acesulfame, acetic acid and propionic acid	HPLC-UV/VIS	Jelly, beverage, tea, juice, coffee, wine, vinegar	0.7–5.0 mg L <sup>-1</sup>	(Le et al., 2017)
Antioxidant and colours	Tocopherols, carotenoids	HPLC-MS and HPLC-DAD	Canola oil, palm fruit, sunflower and olive oils	–2.00 µg mL <sup>-1</sup>	(Flakelar, Prendler, Jackleitner, Howitz, & Doran, 2017)
Preservative	Benzoate	HPLC-UV/VIS	Beverages	0.003–0.004 mg L <sup>-1</sup>	(Pavlovskas-Ilievskis, Velikonja-Marčičevska, & Janžekovič, 2017)
Preservative and colour	Potassium sorbate, sodium benzoate, carmoisine, allura red, ponceau 4R	HPLC-UV/VIS	Energy drink, pomegranate juice and mandarin juice	0.10–0.19 µg mL <sup>-1</sup>	(Apo, Day Zor, & Alsu Dönmez, 2016)
Sweetener	Acesulfame, succharine, cyclamate, sucralose, aspartame	HPLC-MS	Natural water	0.002–0.7 µg L <sup>-1</sup>	(Silas, Boerull, Fontanals, & Marcé, 2015)
Flour treatment	α-amylase	HPLC-MS	Corn flour	NR	(L. Liu et al., 2015)
Anticaking agent	Peroxyanides	HPLC-UV/VIS	Reworked salt, burnt molten salt, refined salt, processed salt, solar salt and other salt	0.02 mg kg <sup>-1</sup>	(Soo L. Kim et al., 2018)
Sweetener, acidity regulator and preservative	Aspartame, saccharin, citric acid and benzoate	HPLC-CAD-UV/VIS	Drinks, diet supplements and tabletop sweeteners	0.08–2.7 µg mL <sup>-1</sup>	(Greenbecka, Baran, Blaszczak, Pijlak, & Szefer, 2014)
Sweetener	Steviol glycosides	HPLC-PLU	Strawberry jam, low-fat milk, soft drink, yogurt and mixture of steviol glycosides	0.05 nmol	(Barbholomes, Struyf, Luywers, Ceunen, & Geuns, 2016)
Preservative	Benzoate	HPLC-UV/VIS	Cheese and yogurts	0.526 mg Kg <sup>-1</sup>	(Zamanl Moadet et al., 2017)
Acidity regulator	Citric acid and malic acid	HPLC-MS	Agriroot liqueur	NR	(Akamatsu et al., 2017)
Antifouling agent	Mineral oils	LC-CC-RID	Biogenic olefins	5 mg kg <sup>-1</sup>	(Nemola & Schmidt, 2017)
Acidity regulator, anticaking agent, emulsifying salt, firming agent, flour treatment agent, humectant, raising agent, stabilizer and thickener	Phosphate	HPLC-ESI-MS	Grain	25 ppm	(Daouq, Clark, Lapoley, & Pegg, 2017)
Colour, antioxidant and	Carotenoid and tocopherol	HPLC-UV/VIS and PLU	Pea, lentil, bean and chickpea	NR	(Fudhi, Liu, Hernandez, Tsao, & Randsath, 2017)
Colours	Tartrazine, Sunset Yellow, Azorubine, Amaranth, Ponceau 4R, Erythrosine, Allura Red, Indigo Carmine, Brilliant Blue, Fast Green, Brilliant Black	LC-MS	Sugar and gummi confectionary, ice-cream, and chocolate sweets	2–20 µg kg <sup>-1</sup>	(Murti, Oberson, Mischari, & Mizari, 2016)
Acidity regulator and colour	Malic acid, carotenoids and Citric acid	HPLC and GC-MS	Grapefruit pulp	NR	(Zheng, Zhang, Quan, Zheng, & Xi, 2016)
Colour	Riboflavin	LC-MS	Inaifur formula, breakfast cereal, milk and multivitamin	0.7 mg kg <sup>-1</sup>	(Lee, Song, Sim, & Kim, 2016)
Acidity regulator, anticaking agent, emulsifying salt, firming agent, flour treatment agent, humectant, raising agent, stabilizer and thickener	Phosphate	CE-UV/VIS	Fish and shrimp muscles	0.23–0.33 mg g <sup>-1</sup>	(L. Wang, Li, & Zhang, 2015)
Antioxidant	BHA, BHT and TBHQ	GC-MS	Oil	0.08–0.10 ng g <sup>-1</sup>	(Cacho, Campillo, Villaw, & Hernández-Gordoba, 2016)
Antioxidant	Tocopherol	HPLC-UV/VIS	Palm oil	100.28–175.24 µg L <sup>-1</sup>	(Xia, Gan, Morris, & Ng, 2016)
Antioxidant	PG and BHA	HPLC-UV/VIS	Foodstuff	0.14–0.12 mg L <sup>-1</sup>	(Mironos, Vera, Dies-pascual, Paz, & Andrés, 2017)
Antioxidant	BHA and BHT	GC-MS	Oil	0.0006–0.0012 mg kg <sup>-1</sup>	(Fang et al., 2017)

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Table 2 (continued)

Spectroscopy		Food additives	Detector	Samples	LD	Ref.
Classes		Mannitol	HPLC-UV/VIS	Solid-state fermented rice	15.6 g mL <sup>-1</sup>	(Chiang et al., 2017)
Anoaking agent, humectant, bulking agent, stabilizer, sweetener and sweetener		Sucralose	HPLC-ELSD	Commercial sucralose	0.8–2.0 µg mL <sup>-1</sup>	(Van et al., 2016)
Glazing agent		Mineral oil	L.C-GC-UV/VIS	Dry pasta, bread, biccuit and plum-cake	0.03 mg kg <sup>-1</sup>	(Moret, Scolaro, Bapp, Purcaro, & Conti, 2016)
Electroanalytical						
Classes		Food additives	Working electrode	Samples	LD	Ref.
Acidity regulator, antioxidant preservative and colour retention agent		Ascorbic acid/nitrite	GCE/QND-oNP or thionine	Orange juice	0.34–0.82 µM	(Barcelomé & Fragozo, 2017)
Preservative and colour retention agent		Nitrite	GCE-Pd/Fe <sub>3</sub> O <sub>4</sub> /polyDOPA/RGO	Water and sausage extract	0.5 µM	(Zhao, Xia, Liu, Huang, & Ye, 2017)
Anoaking agent, humectant, bulking agent, stabilizer, sweetener and sweetener		Mannitol	AuNP / RGO-GCE	Sugarcane vinasse	7.7 × 10 <sup>-19</sup> mol L <sup>-1</sup>	(Behnamini, da Silva, Sedenho, & Stadiotto, 2017)
Flour treatment agent, preservative, bleaching agent and antioxidant		Sulfite	Screen-printed carbon	Wine	0.4 mg L <sup>-1</sup>	(Ramos, Gonçalves, Vystoučil, & Rodrigues, 2016)
Acidity regulator		Malic acid	Screen-printed carbon	Wine	1.89 µM	(Moliner-Abad, Alonso-Lomillo, Dominguez-Renedo, & Arco-Murín, 2015)
Preservative		Sulphur dioxide	Screen-printed gold nanoparticles mixed with silver	Wine	NR	(Schneider, Turke, Fischer, & Klumartin, 2014)
Colour and preservative and colour retention agent		Amaranth and nitrite	CPE, CuO / SWCNTs	Orange juice, apple juice and sausage	0.001–0.5 µM	(Bijad, Karimi-Molahi, Farsi, & Shahidi, 2017)
Flavour enhancer		L-Glutamate	CEBMG	NR	4.4 10 <sup>-6</sup> M	(Doroshko, Korotkova, Shabueva, & Mosolov, 2015)
Flavour enhancer		Sodium glutamate	MB-Screen-printed carbon	Unspiced beef	3 µM	(Hughes, Pemberton, Padden, & Hart, 2015)
Acidity regulator, anoaking agent, emulsifying salt, firming agent, flour treatment agent, humectant, raising agent, stabilizer and thickener		Phosphate	Screen-printed	Water	4 µM	(Gini, Talarico, Pallacchi, Moscone, & Ardini, 2016)
Acidity regulator, anoaking agent, emulsifying salt, firming agent, flour treatment agent, humectant, raising agent, stabilizer and thickener		Phosphate	Au electrode	Seawater	0.05 mmol L <sup>-1</sup>	(Buras, Romanyasi, Strižbig, & Garçon, 2016)
Antioxidant		TBHQ and BHA	GCE-MIP/MWCNT	Oil and margarine, mayonnaise	0.85–0.50 mmol L <sup>-1</sup>	(Moretz, Oliveira, Sched, Dall'Agnola, & Turley, 2016)
Colour		Sunset yellow	GCE/GO and MGO	Mirinda drink and minute milk	2.0x10 <sup>-9</sup> mol L <sup>-1</sup>	(Li et al., 2016)
Colour		Ponceau 4R	e-MnO <sub>2</sub> microspheres/GC/GCE	Soft drink	0.1 nmol dm <sup>-3</sup>	(Huang, Zeng, & Wang, 2016)
Colour		Sunset yellow and tartrazine	Au-Pd-RGO/GCE	Juice and rehydrant	1.5x10 <sup>-9</sup> mol L <sup>-1</sup>	(Jin Wang et al., 2016)
Colour		Sunset yellow and tartrazine	GCE/AuNRs and ERGO	Orange juice, orange soda and jelly	2.4–8.6 nM	(Deng, Li, Li, & Huang, 2016)
Sweetener		Aspartame	GCE/ZnONPs	Refrigerant and sugar	3.68 µM	(Bajgobind, Kanchi, Sharma, Bhatnagar, & Sabeena, 2016)
Colour		Alura red	GCE/PDPA-Cr-Ni	Strawberry juice	8.0 nmol L <sup>-1</sup>	(Li, Yu, Shi, Yue, & Qi, 2016)
Antioxidant		BHA and PG	Au-Pt nanorods	Oil, potato chips and cookies	0.046–0.024 mg L <sup>-1</sup>	(Wu et al., 2016)
Colour		Indigo carmine	CPE/NBE	Candy coated chocolate, jelly powder and food coloring	0.36 µmol L <sup>-1</sup>	(Arvand, Saberi, Ardabi, & Mo'hammadi, 2017)
Colour		Tartrazine, sunset yellow and carmalum	GCE-MWCNTs	Soft drinks	0.11–0.12 µM	(Sierra-Rosales et al., 2017)
Acidity regulator and antioxidant		Ascorbic acid	Gold	Orange Juice	30 µM	(Kuss & Compton, 2017)

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Table 2 (continued)

Classes	Food additives	Detector	Samples	LD	Ref.
Spectroscopy					
Preservative and colour retention agent	Nitrite	Pt/Pt <sub>2</sub> O <sub>3</sub> /poly(DOPA)/RGO modified on GCE	Water and sausage extract	0.5 µM	(Zhao et al., 2017)
Flour treatment agent, preservative, bleaching agent and antioxidant	Sulfite	GCE-AuNPs	Wine	0.045 µmol L <sup>-1</sup>	(Yu, Feng, Chen, Wang, & Jia, 2017)
Acidity regulator	D- and L-lactate	Biosensor array chip with platinum	Inoculum sludge	0.7–0.9 µmol L <sup>-1</sup>	(Pilas, Yazici, Selmer, Kougen, & Schöningh, 2017)
Flour treatment agent, preservative, bleaching agent and antioxidant	Sulfite	Pellet electrode modify graphic	Tomato lechup, jam, sugar, dried grapes and wine	1.7 µM	(Devaramani, Adirakati, & Mahalingappa, 2017)
Flour treatment agent, preservative, bleaching agent, antioxidant	Sulfite	GPE/Au <sup>-</sup> Si <sub>3</sub> N <sub>4</sub> <sup>+</sup> G <sup>-</sup>	White wine and coconut water	0.88 mg L <sup>-1</sup>	(Winkler, Barros, Magosso, & Jose, 2017)
Bleaching agent and antioxidant	Sulfite				
Acidity regulator	L-lactate	PGE/MS/PDDA/AuNP/GOx	Yogurt and fermented milk samples	0.47 µM	(Das et al., 2017)

FAAS = Flame Absorption Atomic Spectroscopy; NIR = Near Infrared; BLU = Fluorescence; AAS = Absorption Atomic Spectroscopy; UV/Vis = Ultraviolet/Visible; IR = Infrared; NMR = Nuclear Magnetic Resonance; FTIR = Fourier Transformed Infrared; DRS = Diffuse Reflectance Spectroscopy; CL = Chemiluminescence; GC = Gas Chromatography; MS = Mass Spectroscopy; HPLC = High Performance Liquid Chromatography; FID = Flame Ionization Detector; CAD = Charged aerosol detection; LC = Liquid Chromatography; EIS = Electrochemical Impedance Spectroscopy; CE = Capillary Electrophoresis; BHA = Butylated hydroxyanisole; BHT = Butylated Hydroxytoluene; TBHQ = Tertiary Butylhydroquinone; PG = Propyl Gallate; EISD = Evaporative Light Scattering Detection; GCE = Glassy Carbon Electrode; CNO-oAP = Small Carbon Nano Onions-Ortho-Aminophenol; RGO = Reduced Graphene Oxide; SWCNTs = Single-Walled Carbon Nanotubes; MG = Modified Gold; MB = Meldola's Blue; MWCNT = Multi Walled Carbon Nanotubes Covalently Bonded (Molecularly Imprinted Poly); MGO = Magnetic Graphene Oxide; ERGO = Electro-reduced Gold Nanorods Decorated Graphene Oxide.

the stationary phase according to the polarity of the desired compound to be separated. Sometimes, auxiliary columns, called guard columns, with the same chemical constitution of the column are used prior to the analytical column (Christian et al., 2014).

In food analysis by HPLC, several detectors, which convert the flux containing the mobile phase and target compounds to an electric signal proportional to its concentration are commonly used. Among these, the UV/Vis absorption detector measures the absorption of radiation by chromophore-containing compounds. A fluorescence detector measures the emitted light. A refractive index detector measures the change in the refraction index of the mobile phase, due to dissolved analytes. Electrochemical detectors are based either on electrochemical oxidation-reduction of the analyte or changes in conductivity of the eluent. The MS detector separates and detects ions in the gas phase and is the most sensitive and selective detector used in HPLC and GC (Christian et al., 2014). The coupling of HPLC and GC with MS detection permit the simultaneous determination of various food additives in foodstuffs (Logue et al., 2017).

The main advantages of HPLC are efficiency, a great diversity of stationary phases, high sensitivity due to different detector types and easy recovery of the samples with a suitable resolution. However, the excessive costs of the instrumentation, the necessity of a specialised operator and the generation of toxic residues from organic solvents are the main disadvantages of HPLC.

Capillary electrophoresis has been indicated as a versatile and high-performance tool that enables fast and efficient separations, with low consumption of samples, solvents and reagents. This technique relies on the electro-migration of the concerned species through a capillary under an electrical field. Coupling capillary electrophoresis separation with HPLC or MS permits suitable sensitivity and selectivity in food analysis (Le et al., 2017).

Table 2 lists recent studies that developed and applied analytical chromatography techniques in the analysis of different classes of food additives in various feedstocks and/or foodstuffs.

### 3.3. Electroanalytical techniques

The last two decades have experienced a considerable increase in the popularity of electroanalytical techniques in food quality control, due to the operational simplicity, low cost and minimum preparation samples steps. These techniques measure the electrical properties, such as current, potential, charge, resistance, conductance, impedance and conductivity, to identify and quantify organic compounds, as the electrical properties are proportional to the target compounds. In food analysis, the electroanalytical techniques that use the applications of potential to promote a reaction of electron transfer following a current measurement, called voltammetric techniques, can be used to identify and quantify food additives in complex samples, such as feedstocks and foodstuffs (Wang, 2001).

In voltammetric techniques, the potential is changed to promote an electron transfer reaction, and the resulting current is recorded as a function of the applied potential, producing a voltammogram that can be used in the identification (peak position) and quantification (peak weight) of the target compounds, but only if this compound is electroactive (reducible or oxidisable). Voltammetric techniques are classified according to the way the potential is imposed on the electrode. Differential pulse voltammetry (DPV) and square wave voltammetry (SWV) are typically used for analytical purposes because the responses are based on the superior elimination of the capacitive/background current, promoting a sensitivity comparable to chromatographic techniques (Scholz, 2010).

In DPV, small pulses of fixed amplitude superimposed on a linear potential ramp are applied in the working electrode where the reaction of interest occurs. The current is measured before and after applying the pulse, producing a resulting signal that is plotted versus the potential applied, and consists of the peak current, with a height directly

proportional to the concentration of the target compound and a peak potential that can be used for its identity (Dahmen, 1986).

In SWV, a waveform composed of a symmetrical square wave, superimposed on a base staircase potential, is applied in the working electrode. The measured current presents excellent sensitivity because the peak currents are measured once at the end of the forward peak and once at the end of the reverse peak, producing a net signal larger than either the forward or reverse signals, consequently generating a sensitivity higher than the DPV (Wang, 2001).

The analytical advantages of DPV and SWV include excellent sensitivity with a broad useful linear concentration range for both inorganic and organic species, a large number of useful solvents and electrolytes, a wide range of temperatures, rapid analysis times (seconds), simultaneous determination of several analytes, and the ability to determine kinetic and mechanistic parameters (Mirceski, Komorsky-Lovric, & Lovric, 2007).

DPV or SWV requires the use of an electrochemical cell constituted by two or three electrodes, called the electric conductor, plus an inert electrolytic solution, called the ionic conductor or supporting electrolyte. This inert electrolytic solution is used to decrease the resistance of the solution, minimise the electro-migration effects and to control the ionic strengths of the medium. Inorganic salts, buffer solutions and mineral acids are frequently used as supporting electrolytes, which can be pure and should not be easily reduced or oxidised in the potential range of the analysis.

The instrumentation consists of two inexpensive integrate circuits, one consists of a polarising circuit that applies the potential between the working and reference electrodes, and one is a measuring circuit that indicates the current that is produced between the auxiliary and working electrodes. Consequently, the instrumentation and the analysis present relatively modest costs and a considerable operational facility, which are the main advantages of the electroanalytical techniques (Wang, 2001).

The change in working electrode promotes alterations in the redox reaction, consequently, altering the current values measured, because the target compounds change their charges at the surface of the electrode, by exchanging one or more electrons with the conductor. In this electrochemical reaction, both the reduced and oxidised compounds remain in solution while the conductor is chemically inert and serves only as a conductor of electrons. The adequate choice of the working electrode depends on the redox behaviour of the target compound and the background current over the potential region used in the analysis. The electrical conductivity, surface reproducibility, potential window, mechanical properties, cost, availability and toxicity, are also important considerations in the implementation of this technique (Scholz, 2010).

Mercury, in the forms of the dropping mercury electrode, hanging dropping mercury electrode and mercury film electrode, was the most used material because it extends the negative potential window, shows high reproducibility, is readily renewable and has a smooth surface. However, in the last two decades, it was substituted by other surfaces, due to the limitation of only cathodic potential regions and high toxicity (Scholz, 2010).

Solid amalgam electrodes, produced from the mixture of a metal powder (e.g., silver, copper, platinum and gold) with mercury liquid following the amalgamation process, presents very close properties to the mercury electrode but without the mercury residues (De Souza, Mascaro, & Fatibello-Filho, 2011). These electrodes are a suitable alternative in the development of sensing devices for food analysis applications.

Solid electrodes composed of carbon and noble metals have been used as working electrodes, adding an extended anodic potential window to monitor oxidisable compounds. However, the analytical responses are dependent on the state of the surface electrode, which requires pre-treatment and polishing steps to obtain suitable responses. Carbon-based electrodes, such as graphite powder with liquid or solid binders, carbon fibres, highly-oriented pyrolytic graphite, carbon

nanotubes and boron-doped diamond, have been evaluated as adequate surfaces in electroanalysis. Platinum and gold electrodes present high analytical responses but a limited range of applications, due to background currents associated with oxide formation on their surfaces (Wang, 2001).

Chemically-modified electrodes represent an important expansion in the applications of the electroanalytical techniques. These electrodes present deliberate surface alterations, to improve the analytical performance and/or resolve many electroanalytical problems, such as sensitivity and selectivity. However, the reproducibility associated with these modifications is the main drawback to its utilisation in food control analysis (Sierra-Rosales, Toledo-Neira, & Squella, 2017; Wang, 2001).

In the last year, the use of electrochemical aptasensors, while are DNA or RNA aptamers with adequate secondary structures that function as ligands in different transduction systems, are capable of binding to a target molecule with high affinity and specificity (Amaya-González, de los Santos-Álvarez, Miranda-Ordieres, & Lobo-Castañón, 2013). The success in the development of an aptasensor involves immobilisation of the aptamer on the transducer surface. Advances in nanotechnology have produced aptasensors with adequate stability and surface coverage by the aptamer while maintaining a high binding affinity in solution, permitting their use in food analysis (Vasilescu, Hayat, Gáspár, & Marty, 2018). The utilisation of porous paper as the substrate for DNA or RNA aptamer immobilisation and construction of an aptasensor for food analysis has also been reported (Vasilescu et al., 2018).

Table 2 highlights recent studies that developed and applied analytical procedures using electroanalytical techniques in the analysis of different classes of food additives in various feedstocks and/or foodstuffs. All these works evaluated analytical protocols to minimise the pre-treatment steps and improve the sensitivity and selectivity of the food analysis.

#### 4. Perspectives in food additives analysis

This review has shown the main food additives classes employed in the processing, packing and storage steps of foodstuffs, indicating the principal compounds and their specific applications. The analytical techniques used by the food industries in the identification and quantification of these food additives were also presented, indicating the advantages and disadvantages of each approach, to supply the necessity of rigorous analytical control and, in this way, ensure a suitable quality and safe food.

In addition, the article presented the literature published in the last 10 years (Figs. 2–4) in which spectroscopy, chromatography and electroanalytical techniques were developed and applied to food additives analysis. Each method presents specific characteristics, including sensitivity, selectivity, analysis costs, efficiency and operational facility, which include minimum sample preparation and ease in instrumentation use.

Nonetheless, the improvement in the quality of the analytical methods and a decrease in the operational costs still need to be considered for direct applications in the quality control by the food industries. In general, in food industries, the chemical composition of the samples is known, and so selectivity is not always the fundamental factor in the choice of the analytical technique. Meanwhile, operational costs, facility in the sample preparations and efficiency in the analysis are essential factors in food quality control laboratories.

The spectroscopic techniques require little or no preparations steps of the sample and present operational facility. However, these techniques have poor selectivity, because all food additives present functional chemical groups that absorb or emit light, and, therefore, cannot be correctly identified using spectroscopic techniques. Although, the use of artificial neural networks can improve the selectivity of food analysis.

The chromatographic techniques promote an adequate separation, identification and quantification of the different classes of food

additives. However, their use demands complex pre-treatment of the food samples and high technical skills by the analyst. Besides, their use in the food industries increases the operational cost in the quality control because of the high maintenance cost, related to employment of high-purity standards and reference reagents, and specific columns. Moreover, the use of chromatographic techniques in food analysis produces toxic residues from organic solvents employed in the analysis.

In the LC techniques, the development of columns specific to food additives classes and the search for mobile phases with low toxicity and low cost are fundamentals factors to be considered in food analysis. In GC techniques, the derivatization reactions required to convert the food additives to volatile and thermally stable compounds could be improved to expand the use of these techniques in food analysis control.

The electroanalytical techniques employed require the food additive to present electroactivity (reduction and/or oxidation reactions) at the electrode surface, producing current that is used in its quantifications. These techniques exhibit high sensitivity, operational facility, are fast, simple and low cost. Many materials have been developed to improve the selectivity and thereby enhance the use of the electroanalytical techniques in food quality control. According to Fig. 4, the food additives most commonly evaluated by electroanalytical methods are antioxidants, sweeteners and colourants. The improvement in the pre-treatment of the electrode surface and the development of a material with a homogeneous surface, with respect to electrochemical activity, are fundamentals to extend the applicability of electroanalytical techniques in food control analysis.

Development of a specific “fingerprint”, an apparatus able to mimic the human olfactory perception, based on electronic nose measure and an appropriate algorithm, could permit the establishment of the regression models between electronic nose signals and food additives.

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