

ADOPTED: 26 January 2022

doi: 10.2903/j.efsa.2022.7136

Safety assessment of the substance fatty acid-coated nano precipitated calcium carbonate for use in plastic food contact materials

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Abstract

The EFSA Panel on Food Contact Materials, Enzymes and Processing Aids (CEP) assessed the safety of the substance identified by the applicant as 'fatty acid-coated nano precipitated calcium carbonate'. It is intended to be used as a filler in all plastics at up to 5% for contact with acidic food and at up to 40% for contact with all other types of food. Articles made with the substance are intended for long-term storage over 6 months at room temperature and below. No information was provided on the mechanism of coating as to whether there is chemical modification of the surface and/or physical adsorption. The substance was not properly characterised, either as pristine material or when incorporated into plastic. Contrary to the non-coated material, data show that the coated material does not dissolve fully and quickly under pH conditions simulating gastrointestinal tract. Surface analysis with electron microscopy of low-density polyethylene (LDPE) samples before and after migration experiments showed major differences that indicate release of the substance from the surface after the tests with 3% acetic acid and to a lesser extent with 10% ethanol. This is consistent with measurable migrations that were up to 39 mg CaCO₃/kg when using an LDPE sample made with 5% of the substance in contact with acetic acid for 64 days at 40°C. The required data on the release of nanoparticles and on the potential toxicity of the substance in nanoform were not provided. Therefore, the Panel could not conclude on the safe use of the substance.

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Keywords: calcium carbonate, coated, fatty acids, nano, filler, food contact materials, safety assessment

Requestor: Federal public service of the Health, Food Chain Safety and Environment, Belgium

Question number: EFSA-Q-2019-00233

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Competing interests: R. Franz declared that Fraunhofer institute at which he is employed provides advisory services to private business operators active in the sector on food contact materials. In line with EFSA's Policy on Independence (https://www.efsa.europa.eu/sites/default/files/corporate_publications/files/policy_independence.pdf) and the Decision of the Executive Director on Competing Interest Management (https://www.efsa.europa.eu/sites/default/files/corporate_publications/files/competing_interest_management_17.pdf), a waiver was granted to R. Franz regarding his participation to the EFSA's Working Group on Food Contact Materials (FCMWG) in accordance with Article 21 of the Decision of the Executive Director on Competing Interest Management. Pursuant to Article 21(6) of the above-mentioned Decision, the involvement of R. Franz is authorised as a member in the FCM WG, allowing him to take part in the discussions and in the drafting phase of the scientific output, but he is not allowed to be, or act as, a chairman, a vice-chairman or rapporteur of the working group.

Note: The full opinion will be published in accordance with Article 10(6) of Regulation (EC) No 1935/2004 once the decision on confidentiality, in line with Article 20(3) of the Regulation, will be received from the European Commission. The following information have been provided under confidentiality and they are redacted awaiting the decision of the Commission: the manufacturing details, the identities and levels of impurities and some of the information on the particle and agglomerate sizes.

Legal notice: The full opinion will be published in accordance with Article 12 of Regulation (EC) No 1331/2008 once the decision on confidentiality will be received from the European Commission).

Declarations of interest: The declarations of interest of all scientific experts active in EFSA's work are available at <https://ess.efsa.europa.eu/doi/doiweb/doisearch>.

Suggested citation: EFSA CEF Panel (EFSA Panel on Food Contact Materials, Enzymes and Processing Aids), Lambré C, Barat Baviera JM, Bolognesi C, Chesson A, Coconcelli PS, Crebelli R, Gott DM, Grob K, Lampi E, Mengelers M, Mortensen A, Steffensen I-L, Tlustos C, Van Loveren H, Vernis L, Zorn H, Castle L, Di Consiglio E, Franz R, Hellwig N, Milana MR, Merkel S, Barthélémy E and Rivière G, 2022. Scientific Opinion on the safety assessment of the substance fatty acid-coated nano precipitated calcium carbonate for use in plastic food contact materials. EFSA Journal 2022;20(2):7136, 8 pp. <https://doi.org/10.2903/j.efsa.2022.7136>

ISSN: 1831-4732

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The EFSA Journal is a publication of the European Food Safety Authority, a European agency funded by the European Union.



† Deceased.

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1. Introduction

1.1. Background and Terms of Reference as provided by the requestor

Before a substance is authorised to be used in food contact materials (FCM) and is included in a positive list, EFSA's opinion on its safety is required. This procedure has been established in Articles 8, 9 and 10 of Regulation (EC) No 1935/2004¹ of the European Parliament and of the Council of 27 October 2004 on materials and articles intended to come into contact with food.

According to this procedure, the industry submits applications to the Member States' competent authorities which transmit the applications to the European Food Safety Authority (EFSA) for their evaluation.

In this case, EFSA received an application from the federal public service of the Health, Food Chain Safety and Environment, Belgium, requesting the evaluation of the substance fatty acid-coated nano precipitated calcium carbonate. The dossier was submitted on behalf of the Calcium Carbonate Association Europe (CCA Aisbl).

According to Regulation (EC) No 1935/2004 of the European Parliament and of the Council on materials and articles intended to come into contact with food, EFSA is asked to carry out an assessment of the risks related to the intended use of the substance and to deliver a scientific opinion.

2. Data and methodologies

2.1. Data

The applicant has submitted a dossier in support of its application for the authorisation of the substance fatty acid (saturated and unsaturated)-coated nano precipitated calcium carbonate to be used in plastic food contact materials.

Additional information was provided by the applicant during the assessment process in response to the request from EFSA sent on 30 January 2020, with addendum sent on 4 May 2021 (see Documentation provided to EFSA').

Data submitted and used for the evaluation are:

Non-toxicological data and information

- Identity and characterisation of the particles
- Description of manufacturing process of substance/FCM
- Physical and chemical properties including solubility and dissolution rate tests
- Intended uses
- Existing authorisation(s)
- Migration of the substance from low-density polyethylene (LDPE)
- Surface analysis of a LDPE substance composite before and after migration testing

Toxicological data

- Review of toxicological studies on calcium carbonate (bulk or nano form) and on the fatty acids

2.2. Methodologies

The assessment was conducted in line with the principles laid down in Regulation (EC) No 1935/2004 on materials and articles intended to come into contact with food. This Regulation underlines that applicants may consult the Guidelines of the Scientific Committee on Food (SCF) for the presentation of an application for safety assessment of a substance to be used in FCM prior to its authorisation (European Commission, 2001), including the corresponding data requirements. The dossier that the applicant submitted for evaluation was in line with the SCF guidelines (European Commission, 2001).

The methodology is based on the characterisation of the substance that is/are the subject of the request for safety assessment prior to authorisation, its impurities and reaction and degradation products, the evaluation of the exposure to those substances through migration and the definition of minimum sets of toxicity data required for safety assessment.

¹ Regulation (EC) No 1935/2004 of the European parliament and of the council of 27 October 2004 on materials and articles intended to come into contact with food and repealing Directives 80/590/EEC and 89/109/EEC. OJ L 338, 13.11.2004,p. 4–17.

To establish the safety from ingestion of migrating substances, the toxicological data indicating the potential hazard and the likely human exposure data need to be combined. Exposure is estimated from studies on migration into food or food simulants and considering that a person may consume daily up to 1 kg of food in contact with the relevant FCM.

As a general rule, the greater the exposure through migration, the more toxicological data is required for the safety assessment of a substance. Currently there are three tiers with different thresholds triggering the need for more toxicological information as follows:

- a) In case of high migration (i.e. 5–60 mg/kg food), an extensive data set is needed.
- b) In case of migration between 0.05 and 5 mg/kg food, a reduced data set may suffice.
- c) In case of low migration (i.e. < 0.05 mg/kg food), only a limited data set is needed.

More detailed information on the required data is available in the SCF guidelines (European Commission, 2001).

The assessment was conducted in line with the principles described in the EFSA Guidance on transparency in the scientific aspects of risk assessment (EFSA, 2009) and considering the relevant guidance from the EFSA Scientific Committee, such as the Guidance on risk assessment of the application of nanoscience and nanotechnologies in the food and feed chain: Part 1, human and animal health (EFSA Scientific Committee, 2018). The update of the Guidance on risk assessment of nanomaterials to be applied in the food and feed chain: human and animal health and the Guidance on technical requirements for regulated food and feed product applications to establish the presence of small particles including nanoparticles (EFSA Scientific Committee, 2021a,b) were not published at the moment of the submission of the data by the applicant. They were considered during the assessment, but not for rejecting data compliant with the version from 2018 and not with the versions from 2021.

3. Assessment

According to the applicant², the substance fatty acid-coated nano precipitated calcium carbonate is intended to be used as a filler in all types of polymers to impart strength and rigidity to the polymer, at concentrations up to 5% for contact with acidic food and up to 40% for contact with all other types of food. Articles made with the substance are intended for long-term storage over 6 months between –10°C and +30°C.

The substance was not evaluated by SCF and EFSA in the past. However, the bulk calcium carbonate (not in nanoform) and all the fatty acids used to coat the particles are authorised and listed in the Regulation (EU) No 10/2011 under either a single substance entry or a mixture. None of the calcium carbonate (FCM No. 21, calcium salts of carbonic acid) and the fatty acids have a restriction. Calcium carbonate is also authorised as food additive (E 170) and was re-evaluated by the ANS Panel in 2011 (EFSA ANS Panel, 2011).

3.1. Non-toxicological data

3.1.1. Identity of the substance³

According to the applicant, the substance is a white powder in the form of nanoparticles [REDACTED]

[REDACTED] The amounts of fatty acids added individually or in a mixture to the calcium carbonate are between 0.1% and 5% (w/w). According to the applicant, the fatty acids are aliphatic, linear, C12–C18 saturated and C16–C18 unsaturated. The Panel noted that minor fatty acids may be present in the source material. No information was provided on the mechanism of coating as to whether there is chemical modification of the surface (e.g. formation of fatty acids salts) and/or physical adsorption.

The formula weight for CaCO₃ is 100 Da and for fatty acids ranges from 200.3 Da (lauric acid) up to 284.5 Da (stearic acid).

The purity of the additive is stated to be from 96.5% to 100% (typically 97.5%). However, this was not consistent amongst the set of information provided (e.g. 85–100%, typically 95%). The main impurities in the calcium carbonate are [REDACTED]

² Technical dossier/2subm211019/Appendix B/Sections 1 and 4.

³ Technical dossier/2subm211019/Appendix B/sections 1 and 2; technical dossier/add data Sept 21/supporting letter; related annexes.

Although requested, no information was provided on possible impurities of the fatty acids.

The total surface area of the particles reported in the 'Chemical Safety Report' was 15–30 m²/g mass-specific surface area (MSSA), measured using the Brunauer, Emmett and Teller (BET) method. The particles are not porous, but they form agglomerates that have some porosity.

According to two scanning electron microscopy (SEM) images, the material consists of agglomerated primary particles that are quasi-spherical with a reported diameter of about [REDACTED]

The size of the agglomerates is reported to be around [REDACTED]. For two samples, laser diffraction (LD) determined diameters of up to [REDACTED] and D50 of [REDACTED] respectively. The identity of the tested samples was not clear and the method performance characteristics and data conversion were not provided.

For the characterisation of the size distribution of the primary particles, the applicant provided two sets of information, a 'Chemical Safety Report' and a LD analysis report. According to the 'Chemical Safety Report', the particle size (D50) ranges between 40 and 90 nm boundary composition. However, it was not supported by raw data, method of analysis or data conversion. According to the LD analysis, the particle size ranges from [REDACTED] with a D50 of [REDACTED] and a D98 of [REDACTED]. The identity of the tested samples was not clear, and the method performance characteristics and data conversion were not provided. Therefore, in accordance with the Opinions of the Scientific Committee on nanoparticle assessment (EFSA Scientific Committee, 2018, 2021a,b), the substance was considered not to be properly characterised.

3.1.2. Physical and chemical properties of the substance⁴

The bulk calcium carbonate decomposes at temperatures greater than 825°C.

The solubilisation rate of the coated and non-coated nano calcium carbonate was tested at 37°C, both at pH 3 and 7 and at five time points (0, 30, 60, 90 and 120 min), based on an ultrafiltration test using a filter of 100 kDa pore size. Measurements of the dissolution rate were performed with 100 mg/L of the substance, corresponding to 40 mg/L of calcium. The results show that the nanoparticles do not dissolve completely and rapidly even at pH 3. After 30 min, 18% (corresponding to 7 mg/L) was dissolved at pH 3 and 5% (corresponding to 2 mg/L) at pH 7. After 120 min at pH 3, 23% was solubilised. From this data, the Panel concluded that the fatty acid-coated nano calcium carbonate is much less soluble than the non-coated nano calcium carbonate (95–98% after 30 min at pH 3). The thickness and the percentage of coating of the tested samples were not provided.

In accordance with the EFSA Scientific Committee Opinions (EFSA Scientific Committee, 2021a,b), it is concluded that the substance does not dissolve fully and quickly. Therefore, an assessment at the nanoscale was required, but was not provided although requested.

3.1.3. Characterisation and quantification of the substance after incorporation in FCM⁵

The external surface of the LDPE samples containing the substance were analysed with Electron Microscopy (backscattered SEM, SEM-energy-dispersive X-ray (EDX), and semi-quantitative SEM-EDX) before and after migration experiments lasting for 10 days at 60°C. After contact with 95% ethanol and isooctane, no significant differences were found. However, major differences, such as changes in the shape of agglomerates and the appearance of voids that indicate release of the substance from the surface were observed after the tests with 3% acetic acid and to a lesser extent with 10% ethanol. These observations are consistent with clearly measurable migration observed in 3% acetic acid and 10% ethanol (see Section 3.1.4 below).

The characterisation of the particle size distribution within the plastic by surface analysis of the cross-section was requested. In response, surface analyses of the bulk of LDPE samples containing the substance were performed, using the above-mentioned methodology before and after a migration experiment with 10% ethanol for 10 days at 60°C. Pictures show that the substance is evenly distributed throughout the selected area of the LDPE, with agglomerates in the µm range. Considering 200 primary particles, the average particle size was 73 nm (40–150 nm) before exposure and 74 nm (30–160 nm) after exposure, i.e. showing no significant difference. The images did not show whether

⁴ Technical dossier/2subm211019/Appendix B/section 2; technical dossier/add data Sept 21/supporting letter; related annexes.

⁵ Technical dossier/2subm211019/Appendix B/sections 2 and 5; technical dossier/add data Sept 21/supporting letter; related annexes.

or not the particles were fully embedded in the host polymer, for which cross section images would need to show also the physical boundaries of the composites. The thickness and the percentage of coating of the tested sample were not provided.

3.1.4. Specific migration⁶

The specific migration of calcium, expressed as calcium carbonate, from LDPE containing the substance at 5, 10, 20 and 40% into 10% ethanol, 3% acetic acid, 95% ethanol and isooctane was determined by inductively coupled plasma (ICP) with optical emission spectrometry (OES) and mass spectrometry (MS). The conditions were 10 days at 60°C. 3% Acetic acid was also tested for 64 days at 40°C. The surface to volume (S/V) ratio was 6 dm²/kg.

In the experiments carried out with LDPE containing 40% of the substance, the migration was below 0.03 mg/kg in isooctane, equal to 0.03 mg/kg in 95% ethanol, 3.4 mg/kg in 10% ethanol and 2,502 mg/kg in acetic acid (out of the scope of the request). When using LDPE made with 5% of the substance, the maximum intended for contact with acidic foods, the migration was 39 mg/kg.

Since the substance does not dissolve quickly and fully under pH conditions simulating gastrointestinal tract and the migration of the substance in nanoform was not ruled out, the following information were requested, but not provided:

- migration experiments with LDPE determining nanoparticles in simulants;
- data on polar polymers: on the particle size distribution following incorporation of the substance into the polymer(s), on the swelling of the polymers in contact with aqueous and acidic simulants and on the migration (i.e. the amount, the chemical composition and the size distribution of the released nano particles or ionic chemicals);
- data on the potential release of the substance due to mechanical stress or physical degradation (e.g. abrasion), particularly with dry foods, concluding on the amount, chemical composition and size distribution of the particles.

3.2. Toxicological data

Since the substance does not dissolve quickly and fully under pH conditions simulating gastrointestinal tract and the migration of the substance in nanoform was not ruled out, toxicological data in accordance with the Opinion of the Scientific Committee 'Guidance on risk assessment of the application of nanoscience and nanotechnologies in the food and feed chain' (EFSA Scientific Committee, 2018)⁷ were requested. Instead of data on the substance, the applicant provided toxicological data and a review of toxicological studies on calcium carbonate (bulk and nano form) and on the fatty acids, mainly referring to the REACH registration dossiers, that does not address the data requirement from the Scientific Committee. Therefore, no conclusion could be drawn on the potential toxicity of the substance in nanoform.

4. Conclusions

Based on the information provided by the applicant, the CEP Panel concluded that the substance was not properly characterised, either as pristine material or when incorporated into plastic. The substance does not dissolve rapidly and fully under conditions simulating gastrointestinal tract. The required data on the release of nanoparticles and on the potential toxicity of the substance in nanoform were not provided. Therefore, the Panel could not conclude on the safe use of the substance.

5. Documentation provided to EFSA

- 1) Initial dossier. April 2019. Submitted on behalf of CCA Aisbl.
- 2) Additional data. September 2021. Submitted on behalf of CCA Aisbl.

⁶ Technical dossier/2subm211019/Appendix B/section 5; related annexes.

⁷ For new application(s), the EFSA Scientific Committee Opinions on "Guidance on risk assessment of nanomaterials to be applied in the food and feed chain: human and animal health" and "Guidance on technical requirements for regulated food and feed product applications to establish the presence of small particles including nanoparticles" (EFSA Scientific Committee, 2021a,b) should be considered.

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- European Commission, 2001. Guidelines of the Scientific Committee on Food for the presentation of an application for safety assessment of a substance to be used in food contact materials prior to its authorization.

Abbreviations

ANS Panel	EFSA Panel on Food Additives and Nutrient Sources added to Food
BET	Brunauer, Emmett and Teller
CCA Aisbl	Calcium Carbonate Association Europe
CEF Panel	EFSA Panel on Food Contact Materials, Enzymes, Flavourings and Processing Aids
EDX	energy-dispersive X-Ray
FCM	food contact materials
ICP	inductively coupled plasma
LD	laser diffraction
LDPE	low-density polyethylene
MS	mass spectrometry
MSSA	mass-specific surface area
OES	optical emission spectrometry
REACH	registration, evaluation, authorisation and restriction of chemicals
SEM	scanning electron microscopy
SCF	Scientific Committee on Food
S/V	surface to volume
w/w	weight per weight