UNIVERSITÀ DEGLI STUDI DEL PIEMONTE ORIENTALE
“AMEDEO AVOGADRO”
DIPARTIMENTO DI SCIENZE DEL FARMACO

Corso di Laurea Magistrale in Chimica e Tecnologia Farmaceutiche

TESI DI LAUREA

Development of tattoo ink dispersions based on a REACH-compliant carbon black

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INTRODUCTION

1. TATTOOS HISTORY

The word “tattoo” is believed to have two different derivations, each reflecting a unique cultural context: one is from an ancient Tahitian word, “tatau,” which recalls the technical process by which tattoos were made; the other possible derivation is from the Polynesian word “ta,” which reminds of the “tatata” rhythmic sound of the sharp object used to mark the body. James Cook, an explorer who lived around the 18th century, described the Polynesian word as “tattaw” and brought the word and the tattoo art to Europe [Pesapane, 2014].

In many Polynesian cultures, such as Samoan communities, tattooing was a ceremony. Samoan men traditionally received a tattoo called *pe’a* (Figure 1), while women were tattooed on their thighs with a *malu* [Dance, 2019]. Moreover, in the Polynesian culture, the tattooing process was seen as a test for male endurance. When puberty started, the tattoo ceremony began. The tattooing process was intensive and could take weeks to months to be completed [Taliaferro *et al.,* 2012].

Figure 1 - Traditional Samoan pe'a tattoo [Dance, 2019].

While in the Polynesian culture the tattoo had a protective meaning, and a positive asset, in the Western world, during the reign of Emperor Constantine, tattoos were banned after he converted to Christianity. He found them disrespectful to God due to the idea that humans were reflections of Him and could not be modified [Pesapane, 2014].

In the 20th century, a true tattoo mania emerged among the aristocrats. One of the most famous aristocrats with a tattoo was the Empress Elisabeth of Austria, who had an anchor on her shoulder. For the first time, tattooing was seen as body decor and an act of freedom, to manifest individuality [Schmid, 2013].

It seems that Theodore Roosevelt (1858-1919), the 26th president of the United States, had a tattoo on his chest that represented his family crest, however, this is merely internet folklore. Roosevelt indeed had a tattoo, but it is a gunpowder tattoo accidentally auto-inflicted during childhood. Moreover, Roosevelt is connected to tattoo by *Puck,* America's leading humor magazine, which, on April 10, 1912, made a cartoon entitled *The New Tattooed Man: He Makes an Exhibition of Himself,* which portrays Roosevelt's skin covered in tattoos (Figure 2) [Hoenig, 2020].

Figure 2 - THE NEW TATTOED MAN [Hoenig, 2020].

Anyway, establishing with certainty when tattoos were created is challenging because of their complex history, which dates back more than 6,000 years and involves different epochs and social strata. If we go further back in the annals, Cain was the first one to have a tattoo because of the fratricide he had committed [Ellis, 2012].

The oldest tattoo discovered was on the arm of two 500-year-old Egyptian mummies, and not only mummies from Egypt had tattoos. The mummy Otzi, discovered in 1991 in the Otztal Alps along the border of Italy and Austria, displayed geometric tattoos [Highett, 2018] that appear to be a link to healing procedures, potentially serving as a precursor to traditional acupuncture [Learn, 2018].

Tattoos have a story that embraces centuries and seas, from the Mediterranean to the Pacific, and goes on to the Atlantic, gathering different meanings and significance. Historical sources revealed that tattooing has been a global practice since the Neolithic period.

The Maori people were used to get Moko (facial tattoos) to explain their tribal identity (Figure 3); Japanese people used “Irezumi” to symbolize their affiliation to a gang, while in America, Native Americans used tattoos as an index of their way of life [Taliaferro *et al*., 2012].

Figure 3 – Karewa, c. 1890-1920 with Moko tattoo

Around the 1930s, sailors got tattoos to show their strength, as no one could be seen as weak. Commonly, they got tattooed “HOLDFAST” on their fingers and hands or roosters and pigs on their feet, all of these to protect themselves from falling into the ocean [Taliaferro *et al.,* 2012].

Next to voluntary tattoos, branding took its role in tattoo history. Human branding is the process by which an authority mark someone by force. Slaves or criminals were burned with a red-hot iron, and then the wound was black colored, because in this way it would be permanent [Schmid, 2013].

Tattooing served a punitive role in Greco-Roman culture, as it was used to mark prisoners. The Nazis similarly employed this practice to label Jews in concentration camps. Although Irezumi originated as a unifying procedure, it eventually evolved into an expression of love [Taliaferro *et al*., 2012].

The purpose of branding was to indicate the criminal acts committed by an individual clearly, but it also served to marginalize them [Schmid, 2013].

Even if in the past tattoos were connected primarily to tribal and pagan traditions or sacred symbols, and were seen as membership marks, today they assume various meanings, from describing personal characteristics to simply being an accessory.

In some instances, tattoos are used for medical reasons, such as restoring skin pigmentation after reconstructive procedures, such as nipple and areola tattoos after mastectomy or tattooing over scars [Abed *et al*., 2024].

2. REASONS TO GET A TATTOO

An epidemiological study, whether performed in the USA, Europe, or Australia, describes that the youngest are the ones who get tattooed the most. Older generations are less interested in getting tattoos because they associate tattoos with sailors, criminals, and other undesirable groups [Kluger, 2015].

Due to the young age or because of the lack of information about tattoo safety, tattooing and PMU (Permanent Make-Up) are increasing in popularity day by day.

The choice of tattooing is somehow guided by gender. Usually, men have multiple tattoos in exposed areas, while women have their tattoos in more discreet areas, and usually, tattoos are smaller. Instead, permanent makeup is often performed on their faces [Kluger, 2015].

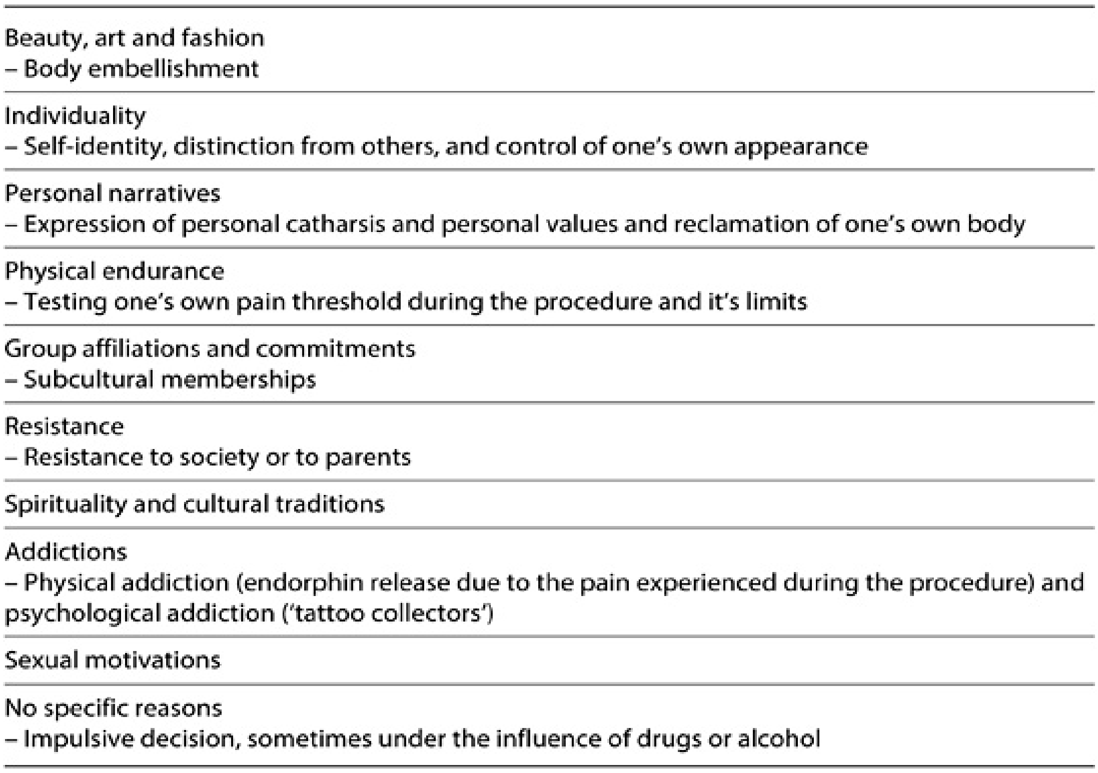
Tattooing is associated with a variety of reasons, most of them psychological:

- self-expression,
- remembrance,
- boosting self-esteem.

Typically, any kind of body modification, such as tattoos or piercings, creates an intimate relationship between the person and his/her body [Stirn *et al*, 2011]. Tattoos contain deeper personal meanings and cannot be reduced to only being a fashion accessory [Wohlrab *et al*., 2007].

According to Wohlrab *et al*., ten categories can summarize the major reasons to get a tattoo (Table 1).

Table 1 – Categories of reasons why to get a tattoo [Kluger, 2015].



Beauty, art and fashion – Body embellishment
Individuality – Self-identity, distinction from others, and control of one's own appearance
Personal narratives – Expression of personal catharsis and personal values and reclamation of one's own body
Physical endurance – Testing one's own pain threshold during the procedure and it's limits
Group affiliations and commitments – Subcultural memberships
Resistance – Resistance to society or to parents
Spirituality and cultural traditions
Addictions – Physical addiction (endorphin release due to the pain experienced during the procedure) and psychological addiction ('tattoo collectors')
Sexual motivations
No specific reasons – Impulsive decision, sometimes under the influence of drugs or alcohol

Understanding the motivations behind acquiring tattoos is necessary to eliminate the negative stigmatization of body modifications [Wohlrab *et al.*, 2007].

Associated with tattoos, there is a social perception. Nontattooed people tend to perceive tattooed individuals as less intelligent, less professional, and more rebellious, while tattooed people consider individuals with tattoos more attractive and intelligent. Even if the stereotypes about tattooed people are slowly decreasing, they still exist in a professional context [Morlock, 2023].

Perceptions of tattoos vary depending on the profession of the wearer. Visible tattoos are often met with discomfort, particularly when worn by doctors, teachers and judges. Due to the disapproval, people in these professions frequently choose to cover their tattoos. Moreover, there is an implicit bias against tattooed women, who tend to be viewed more negatively than tattooed men [Roggenkamp *et al.*, 2017].

3. DEFINITION AND REGULATION OF TATTOOS

What is a tattoo?

The FDA (Food and Drug Administration) regulates tattoo inks and considers them as cosmetics, as defined by section 201(i) of the Federal Food, Drug, and Cosmetic Act (FD&C Act). Tattoos inks can be considered cosmetics due to the fact they are intended to be introduced or applied to the human body to beautify, promote attractiveness, or alter appearance [FD&C Act].

Tattoos in Europe are considered to be in a grey area, as they do not fall under the categories of cosmetics or chemicals. Because of the absence of precise definitions and consequently of regulation, since 2003, the European Commission has made significant changes in regulating tattoos. The attention paid to regulating tattoos and PMU inks reflects a growing awareness of health risks and a commitment to consumer safety.

Unlike cosmetic products, tattoo inks are injected directly into the dermis, where they come into direct contact with blood, lymphatic fluid, and immune cells. The injection of pigments into the human dermis is a topic that manufacturers have neither specifically considered nor considered. This creates a regulatory gap where tattoo ink producers, who focus on formulation rather than pigment development, face significant challenges in conducting comprehensive safety evaluations for each ingredient [Giulbudagian *et al.*, 2020].

This situation highlighted the urgent need for regulations on tattoo ink constituents, ensuring that pigments intended for permanent application in the skin undergo appropriate toxicological assessments.

Prior to 2022, the regulation of tattoo inks in the EU was based on general frameworks such as the General Product Safety Directive (GPSD) (Directive 2001/95/EC), the CLP (Classification, Labeling, and Packaging) (EC No 1272/2008), which addressed labeling requirements, and the REACH Regulation (EC No 1907/2006) which focused on the registration of chemicals (Figure 4) [Giulbudagian *et al.*, 2020].

Figure 4 – Regulation of tattoos and PMU in 2019 [Giulbudagian et al., 2020].

Across the EU, Member States established the Safety Gates system, formerly known as RAPEX (Rapid Alerts System for dangerous non-food products), to facilitate the rapid sharing of information about products that can lead to risks to safety and health. After receiving numerous RAPEX reports concerning tattoo inks, it became evident that mandatory regulations were necessary.

Resolution ResAP(2003)2 on tattoos and permanent make-up identified substances that should be prohibited in tattoo inks and PMU products, specifically carcinogenic, mutagenic, reprotoxic, and sensitizing substances, as well as preservatives and aromatic amines [Resolution ResAP(2003)2].

Although it was a non-binding recommendation, it successfully raised awareness about the health risks associated with tattoos, paving the way for stricter regulations.

ResAP(2003)2 was then replaced by the Council of Europe Regulation Resolution ResAP(2008)1 on requirements and criteria for the safety of tattoos and permanent make-up. The new resolution expanded the list of forbidden substances previously identified in the ResAP(2003)2, including polycyclic aromatic hydrocarbons (PAHs), heavy metals, metalloids, and nonmetals [Kiszla *et al.*, 2023]. Additionally, and established a list of maximum allowable concentrations of impurities [Resolution ResAP(2008)1].

Between the two ResAPs, the REACH Regulations (the Registration, Evaluation, Authorization, and Restriction of Chemicals) were enacted in 2006.

The REACH Regulations aimed:

- to protect human health and the environment,
- to assess the safety of chemicals used in the EU and to ensure free circulation around the EU,
- to evaluate the chemical's toxicity using alternative methods [REACH Regulation 1907/2006].

Based on the recommendations of the Council of Europe, the European Commission adopted Regulation 2020/2081, which amends Annex XVII of the REACH Regulations 1907/2006, in which hazardous substances, including those used in tattoo inks and permanent makeup, are listed.

Technically, REACH is a system specifically designed for single and pure substances used in industrial applications. It has never been applied to products intended for injection, especially those made from low-purity raw materials [Serup, 2023].

Different members contributed to mandate the Regulation 2020/2081:

- European Commission
- European Chemicals Agency (ECHA)
- Italy, Denmark, Norway, and Germany
- RAC – ECHA's Committee for Risk Assessment
- SEAC – ECHA's Committee for Socio-Economic Analysis
- Seven EU Member States (Belgium, France, Germany, the Netherlands, Slovenia, Spain, and Sweden).

Writing the Regulation was a team effort:

1. the European Commission played a key role in regulating tattoo inks by adopting a new regulation based on the scientific contributions of the European Chemicals Agency (ECHA). This regulation establishes a strong framework for ensuring the safety of tattoo products.

2. Italy, Denmark, Norway, and Germany collaborated to assess the risk of chemicals used in tattoo inks and provided technical assistance, to help ECHA write the Annex XV Dossier.
3. Belgium, France, Germany, the Netherlands, Slovenia, Spain, and Sweden had regulations regarding the chemical composition of tattoo ink mixtures and shared their regulatory experiences [Regulation 2020/2081].

Denmark was a key reference point in this scenario because of its extensive tattoo ink registration system, the InkBase system. This electronic database tracks tattoo ink compositions used in the Country since 2018. This study was a crucial benchmark on tattoo use before the EU-REACH was adopted, providing essential data for future research into tattoo-related health risks and adverse reactions and rapid alerts about hazardous substances [Serup *et al*., 2025].

If a dangerous product is found in one EU Country, an alert is sent to all other Countries, and authorities can decide whether to ban, withdraw, or recall the product.

In recent years, RAPEX has recorded multiple alerts on tattoo inks and helped enforce restrictions on hazardous tattoo inks [Regulation 2020/2081].

The new Regulation 2020/2081 was mandated to uniform the regulations across the EU and came into force on January 4, 2022. It was dynamically linked to the EU Cosmetic Regulation 1223/2009 and the Regulation 1272/2008 (CLP Regulation) to guarantee protection for human health, handle future updates of the substances listed in the Annexes and their application to tattoo inks, if any future different classification of hazardous substances will be made [Regulation 2020/2081].

What changed?

The amendment to Annex XVII restricted and banned substances used in different fields in the EU, such as cosmetics, consumer goods, chemicals in industrial products, and tattoo inks. Entry 75 of Annex XVII regulates the substances contained in tattoo inks.

Following an assessment by the European Chemicals Agency (ECHA), over 4,000 chemical substances found in tattoo ink and PMU were restricted. The use of carcinogenic, mutagenic, and reproductive toxicants, as well as skin irritants and sensitizers, and corrosive substances is now prohibited. Specifically, Pigment Blue 15:3 and Pigment Green 7 were banned, while Azo Pigments, such as Red, Orange, and Yellow, were restricted due to their potential to release primary aromatic amines. Heavy metals, such as Nickel, Chromium, Lead, Cadmium, Arsenic, and Cobalt, were limited because of their toxicity and the possibility of causing allergies. PAHs, primarily found in black inks, were reduced due to their linking to cancer risks. Preservative such as benzyl alcohol and isothiazolinones were also limited.

A Maximum Allowed Concentration (MAC) was made, by which most of the restricted substances were fixed to 0.1%, PAHs to 0.0005%, and a limit of 2 ppm for lead and 0.5 ppm for cadmium was imposed.

Moreover, the new regulation established that clear labels were mandatory on ink products. These labels should say “Mixture for use in tattoos or permanent make-up” and include a complete list of ingredients categorized by common ingredient names or the IUPAC name, the CAS and the EC number, a batch number for traceability, and warnings about the presence of allergens as Nickel and Chromium (VI) [Regulation 2020/2081].

Figure 5 shows the categorization of restricted substances in tattoo inks and PMU under REACH.

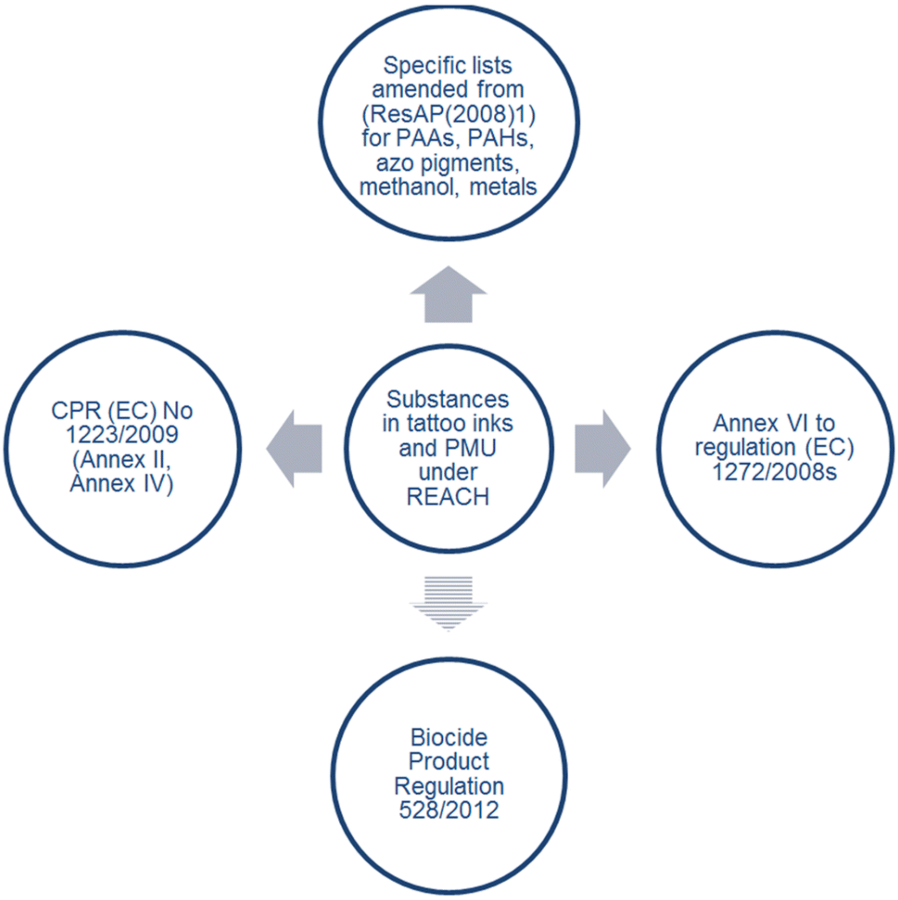
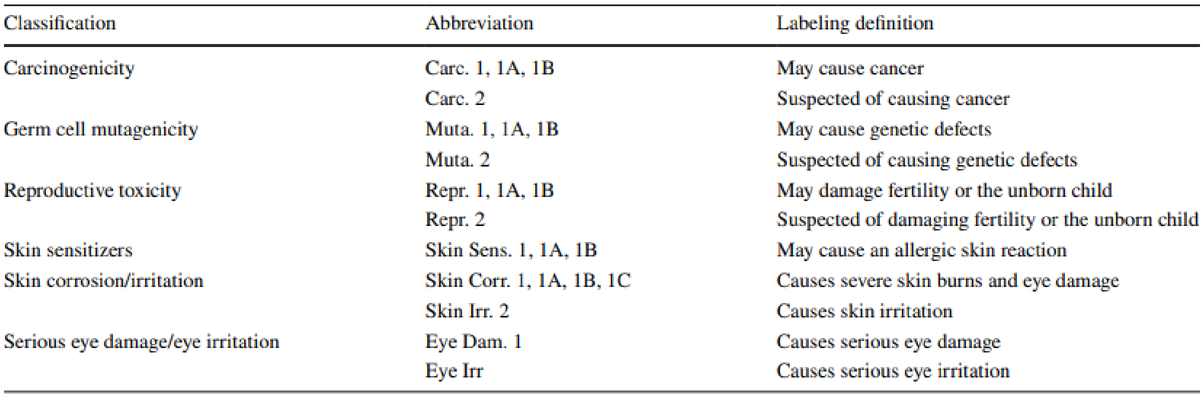


Figure 5 – Substances restriction in tattoo inks and PMU under REACH. Abbreviations used: PAAs, primary aromatic amines; PAHs, polycyclic aromatic hydrocarbons; CPR, cosmetic product regulation [Giulbudagian et al., 2020].

A significant number of substances are regulated under the Cosmetic Product Regulation (CPR) (EC) No. 1223/2009. Since substances are prohibited in cosmetic products, they may also be hazardous if injected into the skin, which could lead to their prohibition in tattoo and PMU inks as well. Annex II of the CPR lists the banned substances, while Annex IV outlines the conditions and concentration limits for colorants allowed in cosmetic products. Instead, Annex VI of the CPL Regulation 1272/2008 restricts substances based on their hazardous classifications (Table 2).

Table 2 – Classification of hazardous categories according to CLP Regulation (EC) 1272/2008 [Giulbudagian et al., 2020].



Classification	Abbreviation	Labeling definition
Carcinogenicity	Carc. 1, 1A, 1B	May cause cancer
	Carc. 2	Suspected of causing cancer
Germ cell mutagenicity	Muta. 1, 1A, 1B	May cause genetic defects
	Muta. 2	Suspected of causing genetic defects
Reproductive toxicity	Repr. 1, 1A, 1B	May damage fertility or the unborn child
	Repr. 2	Suspected of damaging fertility or the unborn child
Skin sensitizers	Skin Sens. 1, 1A, 1B	May cause an allergic skin reaction
Skin corrosion/irritation	Skin Corr. 1, 1A, 1B, 1C	Causes severe skin burns and eye damage
	Skin Irr. 2	Causes skin irritation
Serious eye damage/eye irritation	Eye Dam. 1	Causes serious eye damage
	Eye Irr	Causes serious eye irritation

The ResAP(2008)1 identifies additional groups of banned substances, which include PAHs, aromatic amines, azo pigments, and skin sanitizers. It also encompasses pigments that can produce harmful metabolites such as aromatic amines, toxic metals, or methanol (for its potential eye damage) [Giulbudagian *et al.*, 2020].

Moreover, to protect consumers against infections, stringent regulations were established regarding hygiene and sanitary practices. The regulation related to tattoo inks, which must comply with the Biocidal Product Regulation (BPR) No. 528/2012, sets a list of biocidal products, and none of them were authorized to be used in tattoo inks. Since ResAP(2008)1, sterility requirements were stated and was assured that tattoo inks may contain biocidal products only for preservation after opening, but not to outweigh the poor microbiological quality [Giulbudagian *et al.*, 2020].

Due to the restrictions established by REACH, the ink industry is now facing difficulties because it could not guarantee that over 4,000 chemicals forbidden by REACH were absent or only present in concentrations under 0.00005%, 0.001%, 0.01%, or 0.1%, and undergoes to a significant transformation that can lead to difficulties in enforcement [Serup, 2023].

4. TATTOOS INKS COMPOSITION

Tattoo inks are a complex mixture of various materials, such as pigments that can be organic or inorganic, solvents, and additives like preservatives and binders.

Today tattoo inks dispersion is not only a suspension of a solid in a liquid, but is a high-tech dispersion made from finely spread pigments in a solvent mixture. The ink must be rich in pigments while remaining fluid and dry quickly, with no sedimentation occurring during lengthy tattoo sessions or after extended periods of use [Dirks, 2015].

Figure 6 shows a scheme of the main ingredients into a tattoo ink.

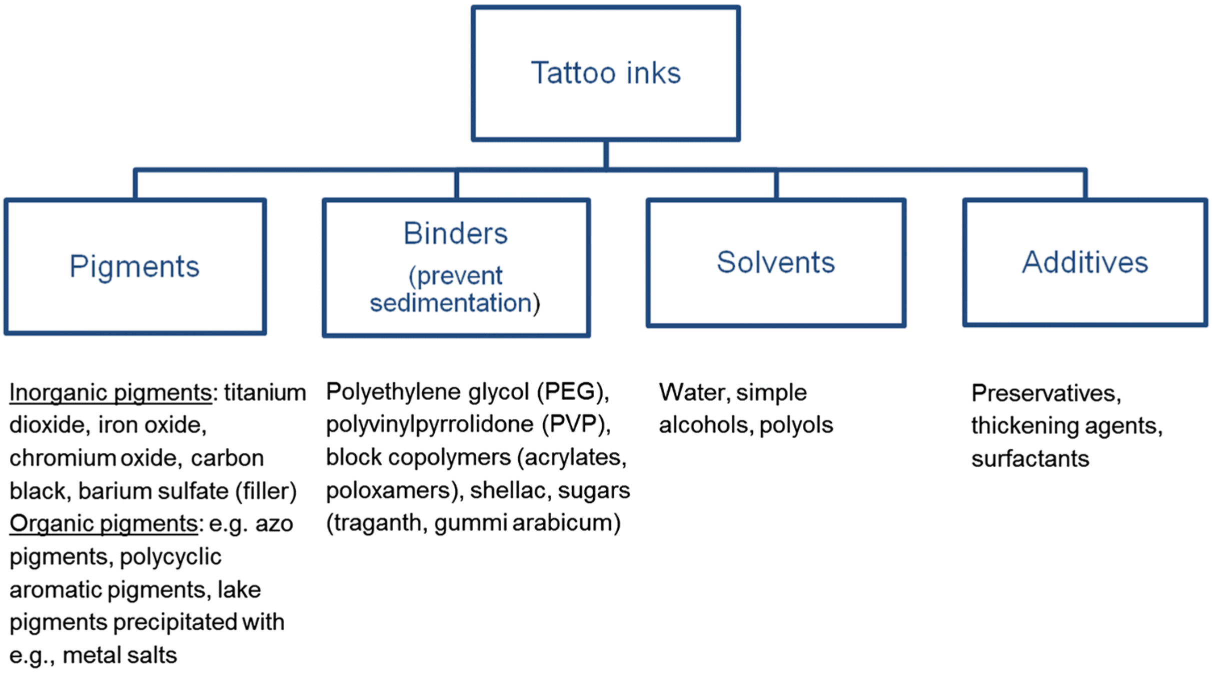


Figure 6: Composition of tattoo inks [Giulbudagian et al., 2020].

Pigments and dyes are defined in DIN 55943, ISO 4617, and ISO 4618. Dye is a soluble colorant, while the pigment is insoluble [Mannari *et al.,* 2015].

In tattoo inks dyes are not used due to solubility, which will cause rapid tattoo degradation [Dirks, 2015].

Figure 7 shows a summary differentiation between different kinds of pigments. The inorganic pigments most used are titanium dioxide, iron oxide, and chromium oxide green. Titanium dioxide pigments are commonly used in tattoos and are of three different types: Rutile, Anatase, and Brookite. The Rutile type is often used in tattoo ink, while the Brookite type has never been used in producing tattoo ink. Iron oxide pigments are limited in tattoo ink, but are used in PMU, while chromium oxide pigments are only used in permanent make-up [Dirks, 2015].

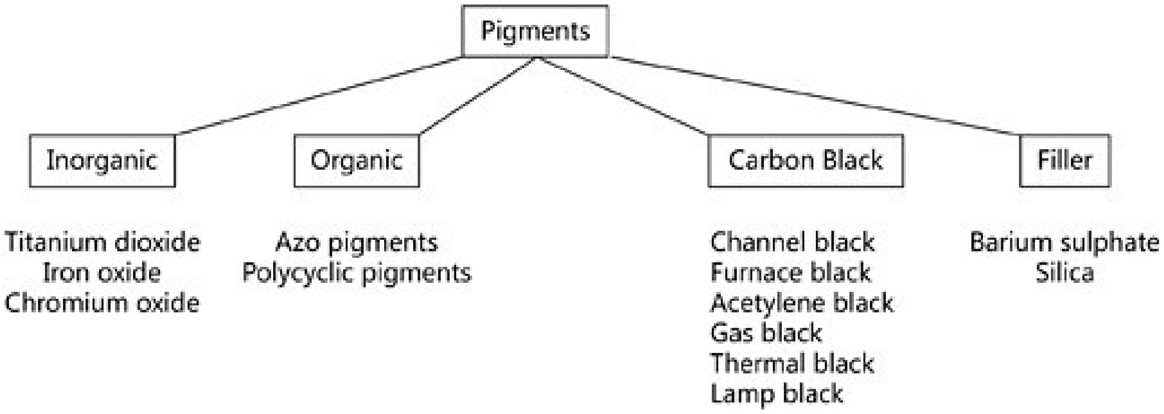


Figure 7 - Division of pigments [Dirks, 2015].

Organic pigments are azo-pigments and polycyclic pigments, which are less dispersible than inorganic and contain aromatic amines as unwanted substances, which are also limited [Dirks, 2015].

Most black pigments are produced by the combustion of hydrocarbons, yielding soot with PAH. The principal constituent of this category is carbon back, which the International Agency of Research in Cancer (IARC) has listed as possibly carcinogenic to humans (category 2B) [Baumler, 2020].

Finally, fillers are inorganic substances added to tattoo inks to enhance storage properties. The most common fillers include barium sulphate and silica [Dirks, 2015].

Along with pigments, other substances are present in a tattoo’s inks, such as preservatives, emulsifiers, and binders. These compounds can lead to adverse reactions and may have mutagenic potential, with some classified by IARC as Group 2A or 2B [Baumler, 2020].

Preservatives are added to tattoo inks to protect them from microbiological spoilage. Since inks are water-based, they can create an environment conducive to the growth of microorganisms. The water activity level of tattoo ink should be kept below 0.6 to inhibit the growth of the microorganisms [Dirks, 2015].

Other additives can be added to the tattoo ink formulations, such as surfactants and thickening agents. Surfactants are added to the tattoo inks to get better dispersion and stabilize pigments during manufacturing, while also helping to extend their shelf life. Thickening agents, instead, are used to increase the viscosity of the ink formula and to prevent the agglomeration and sedimentation of inks during long-term storage [Dirks, 2015].

5. CARBON BLACK

Carbon Black is a pigment commonly used in the cosmetic industry to formulate black products, such as mascara, eyeliner, and eye shadow, and everywhere it is necessary to use a black dye, which is appreciated for its high color stability, chromaticity intensity, and durability. Cosmetic carbon black has an intense black color and does not require any additional substances to achieve this hue.

Due to these characteristics, carbon black is the pigment used to obtain long-lasting black tattoos.

Several industrial processes produce this pigment. Furnace blacks, channel blacks, and gas blacks are the most important ones among the various carbon blacks [Pfaff, 2015].

The size and the dispersibility of the carbon black particles vary depending on their solubility in oil and in water and on the method used to harvest the pigment. At the beginning of the manufacturing process small spherical components known as nodules are observed, with sizes ranging from 15 to 300 nm (Figure 8). These particles aggregate to form larger structures called aggregates, which have a diameter between

85 to 500 nm. When multiple aggregates cluster together, they form larger structures, the agglomerates, with diameters from 1 to 100 μm [Dirks, 2015].

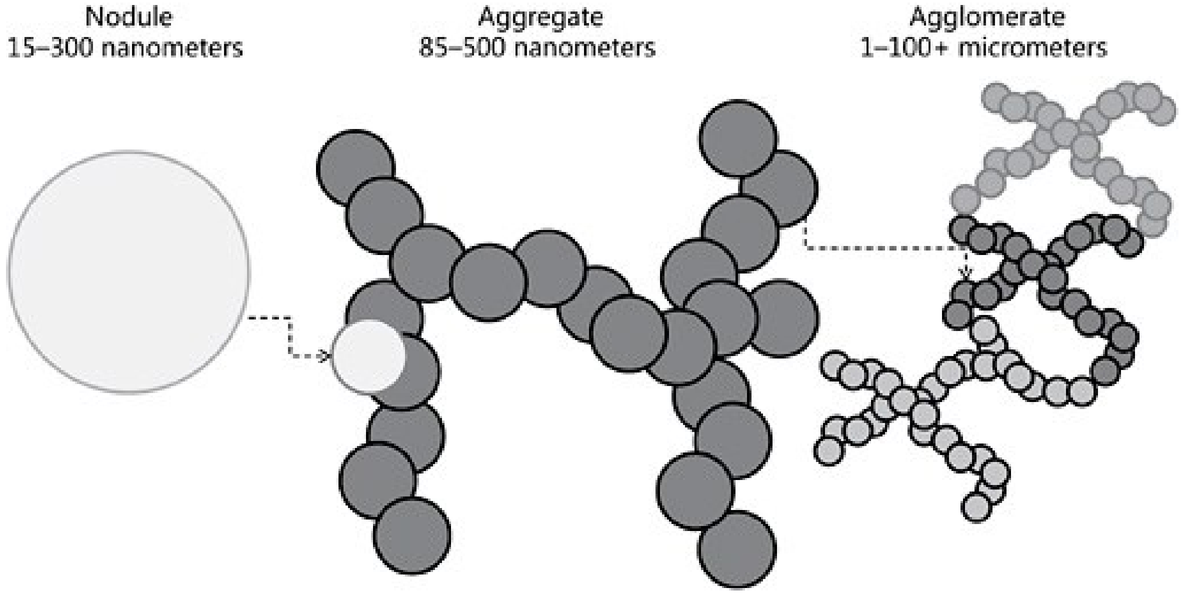


Figure 8 – Carbon black structure evolution [Dirks, 2015].

Carbon black nanoparticles tend to aggregate and interact with cell membranes, destabilizing them and stimulating ROS production (Figure 9). The same effect is produced by lysosomal membrane permeabilization, which can damage the cell. Another effect made by the aggregation of particles is the accumulation of hydrogen peroxide that can lead to H_2O_2-mediated apoptosis [Oh *et al.*, 2025].

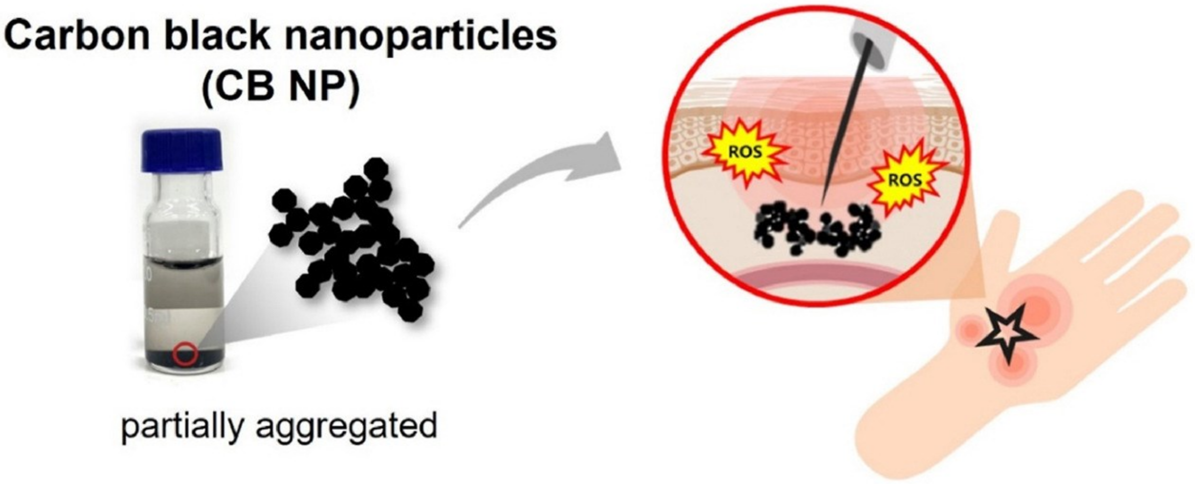


Figure 9 - Illustration of the potential ROS production after injection of carbon black nanoparticles partially aggregated [Oh et al., 2025].

The majority of tattoos are black, with carbon black being the main ingredient used in their ink.

Materials such as chitosan, polyethylene glycol, polysorbates, and Pluronic polymers were considered to enhance the biocompatibility of carbon black. Due to their amphiphilic property, Pluronic polymers are excellent for the dispersion of carbon black. Oh *et al*. developed Pluronic-coated carbon black nanoparticles as biocompatible pigments for tattoo ink, resulting in a dark black dispersion without precipitates, whereas carbon black, without coating, was agglomerated. These tests have shown better dispersion and stability, without cell deaths and oxidative stress (Figure 10) [Oh *et al.,* 2025].

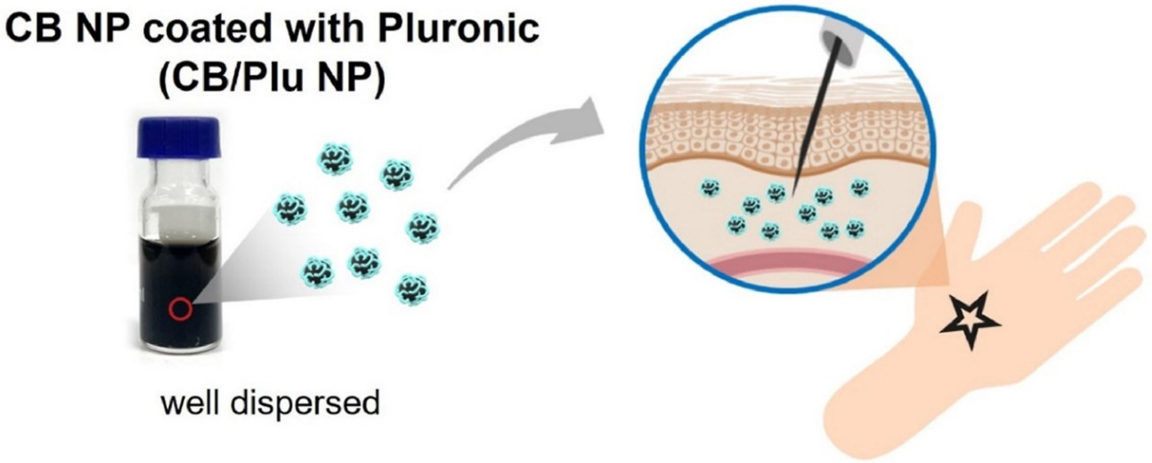


Figure 10 - Illustration of the absence of production of ROS after injection of carbon black nanoparticles, well dispersed, coated with Pluronic polymers [Oh et al., 2025].

6. ADVERSE EFFECTS OF TATTOOING

Tattoo ink has been found to trigger different adverse reactions such as infections, tumors, and inflammatory skin conditions, including eczema, lichen planus, psoriasis, vitiligo and cutaneous sarcoidosis [Mathis *et al*., 2023].

It is uncommon to have an allergic reaction to tattoo pigments due to their encapsulation into the fibrous tissue made by keratinocytes, fibroblasts, and phagocytic macrophages. However, tattooing introduces foreign substances into the skin and may cause an immunologic reaction. In this case, the allergic reaction is associated with one of the many components of the ink, making it difficult to determine which specific ingredient may cause the allergic reaction [Kaur *et al*., 2009].

Skin reactions are primarily associated with red ink, while black inks were previously thought not to provoke adverse reactions. Colboc *et al*. conducted a study to demonstrate the composition of inks and found that 15 patients in their research who had skin reactions to tattoos developed reactions on black inks. Thus, the study suggests that black inks can also provoke skin reactions, probably because of the carbon black and its impurities, most of all the potential allergenic metals within the inks [Colboc *et al*., 2022].

Besides allergic reactions, non-allergic responses, are more common in black inks [Drèno, 2020].

Agglomeration of pigment is typical in black ink and occurs even while the ink is still in the bottle. When injected, the clustering of black pigments happens on the skin, resulting in a larger formation that behaves like a foreign body. These new formations are associated with reactions like papulonodular tattoo response, granuloma formation, autoimmune reactions, the “rush phenomenon” and potentially sarcoidosis [Hutton Carlsen *et al*., 2020].

Over time, when carbon black nanoparticles accumulate in the dermis, sarcoid granulomas form. Black inks are commonly associated with granulomatous reactions, both pigment-bound foreign-body granuloma and sarcoidosis granuloma (Figure 11). Although the two reactions differ due to their cellular involvement, they can coexist in the same individual [Dréno, 2020].

Figure 11 – Sarcoidosis on a black ink tattoo [Dréno 2020].

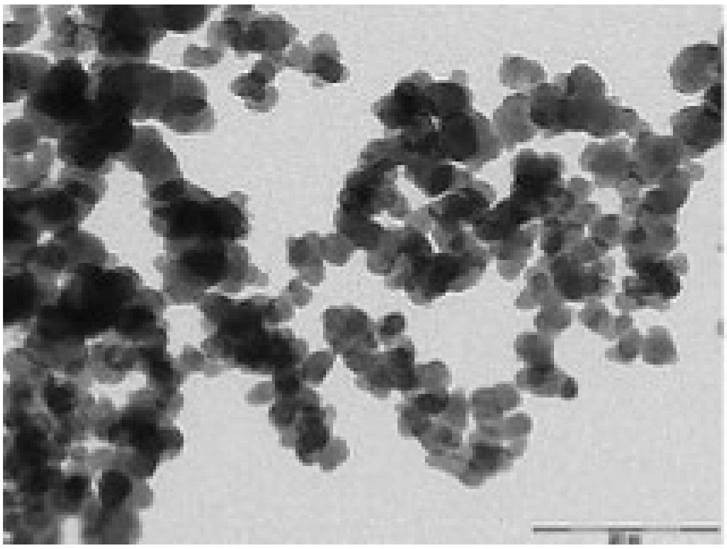


Figure 12 - Carbon black agglomerates [Baumler, 2015].

Figure 12 shows carbon black agglomerates acquired by electron microscopy. These agglomerates can vary the system rheology, influencing how the pigment interacts with the surrounding tissues. As said above, agglomeration can cause adverse reactions, which are a *sine qua non* condition of the particle's agglomeration, and understanding its impact is crucial [Hutton Carlsen *et al.,* 2020].

Another essential aspect to consider when discussing tattoos and their potential adverse reactions is the interaction between tattoo pigments and exposure to UV light.

Over time, UV light can degrade tattoo inks, resulting in the release of toxic substances such as singlet oxygen. Certain inks may produce this ROS due to the surface properties of carbon black derived from PAHs even during laser tattoo removal procedures [Baumler, 2020].

However, the body responds in different ways to tattoo pigments, depending on the tattooed area.

Research has shown that the most tattooed areas are the upper arms, lower arms, legs, back, and shoulders, as seen in Figure 13, which have different characteristics such as skin thickness, blood circulation levels, and sun exposure [Morlock, 2023].

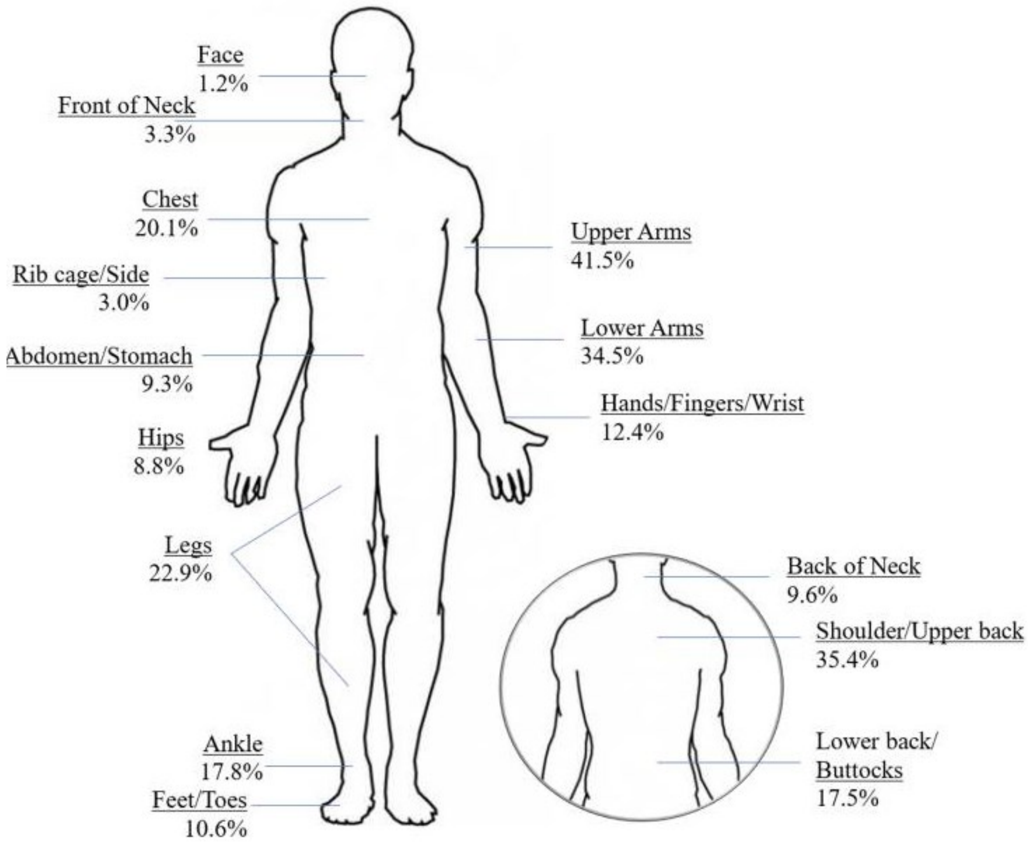


Figure 13: Body areas with major quantities of tattoos [Morlock, 2023].

These factors may influence the adverse effect. For instance, areas with higher blood circulation or major lymphatic drainage networks can lead to a faster pigment migration. In contrast, areas with greater sun exposure may lead to pigment degradation and subsequent inflammatory reactions.

7. SYSTEMIC EXPOSURE TO TATTOO INKS

Tattoo colorants, when injected through needles, get deposited into the intradermal. However, colorants usually don't remain in a specific skin zone. Part of the injected colorant leaves the skin while the wound heals, another part stays in the dermis and carries out the color of the tattoo, and another part is transported through the lymphatic or blood vessel system. In fact, due to their migration, colorants can accumulate in lymph nodes near the tattoo or other organs such as the liver, spleen or kidney [Baumler, 2020].

Inks for tattoos are applied in controlled doses, ensuring long-lasting color on the skin. However, the overall systemic exposure from a typical tattoo is expected to be very low. Currently, there is no valid toxicological model to assess this kind of exposure, and the biokinetic profiles remain largely unknown. As a result, it is not possible to reliably calculate and apply theoretical safety assessments, such as the margin of safety to this issue [Serup, 2023].

Despite the absence of a toxicological model for ink exposure, during tattooing, insoluble pigments and soluble additives are introduced into the dermis and can migrate to lymph nodes and more distant organs, so long-term pigment exposure must be considered. Exposure is determined by the ink quantity applied per surface area of the tattooed skin. Multiple studies have been executed to assess the exposure levels, including measuring the weight of *ex vivo* skin before and after tattooing and quantifying pigments in tattooed *ex vivo* skin [Kochs *et al.*, 2025].

Kochs *et al.* conducted a study using an *in vivo* human quasi-experimental design to assess the worst-case exposure scenarios related to soluble tattoo ink ingredients and their excretion following tattooing. The study by introducing tracer substances into the tattoo inks and tracking their presence in blood, urine and consumable products, suggested that previous regulatory estimates may have been overvalued. Moreover, skin metabolism might detoxify certain hazardous substances, thereby altering their potential toxicity [Kochs *et al.*, 2025].

But, although skin metabolism may help detoxify specific ink components, external factors, like UV radiation, can generate new reactive species, potentially affecting long-term safety.

8. TATTOO REMOVAL

As the popularity of tattoos rose from 2004 to 2018, a subsequent decrease was seen in 2019, while at the same time, an interesting increase in tattoo removal was observed [Kluger, 2025].

Early tattoo removal techniques involved cryotherapy, surgical excision, and skin resurfacing methods such as dermabrasion or chemical peeling. Cryotherapy and surgical removal often result in residual textural changes or scarring, they may also be impractical for larger tattoos. Skin resurfacing only penetrates the papillary dermis, which makes it suitable only for superficial amateur tattoos but ineffective for removing deeper, professionally inked tattoos. Today, lasers are widely used for tattoo removal because they specifically target the pigment, minimizing damage to the surrounding skin. This approach reduces the risk of scarring or dyspigmentation. Laser tattoo removal operates by breaking down pigment particles through both photothermal and photomechanical effects. Essentially, the heat and shockwaves generated by thermal expansion decrease the size of the pigment particles, allowing them to be cleared from the body by macrophages through phagocytosis. However, this process can trigger an immune response from macrophages, leading to hypersensitivity reactions such as erythema, edema, itching, hives, or, in rare cases, anaphylaxis [Hohman *et al.*, 2025].

The procedure does not come without some collateral effects, such as pain, bleeding, infection, burning, blistering, or scarring.

Anyway, in the meantime, the tattoo industry evolves, facing new difficulties, ongoing research and regulations, which will role the balance between artist freedom and consumer health. Whether through improved ink formulations or more advanced removal techniques, the future of tattooing lies in innovation, ensuring that body art will stay both an aesthetic and a responsible choice.

EXPERIMENTAL PART

AIM OF THE WORK

Ink formulations must comply with Regulation 2020/2081, which amends Annex XVII of the REACH Regulation 1907/2006 and became effective on January 4, 2022 [Regulation 2020/2081].

This research aimed to obtain a carbon black dispersion for tattoo inks as closely as possible to the existing formulations while containing reduced levels of hazardous substances.

The final objective was to create a highly effective black ink that was capable of migration yet remained fixed, did not change color over time and was characterized by concentrations of polycyclic aromatic hydrocarbons (PAHs), heavy metals, and aromatic amines within acceptable ranges.

The initial step involved selecting among carbon blacks available on the market the one that met the REACH Regulation limits. Subsequently, the experimental work continued with identifying and testing appropriate ingredients, such as wetting agents and dispersants, to produce carbon black dispersions suitable for use in tattoo inks.

MATERIALS

All information about raw materials used in this experimental work was taken from company documents (Technical Data Sheet).

PIGMENT

- CARBON BLACK DIABOLIK CI 77266

  INCI NAME: CI 77266

  CAS Number: 1333-86-4

  Chemical and physical properties: it is a black pigment of mineral origin. It looks like a fine, almost impalpable powder, with intense clack color and no odor. The powder is water insoluble, but it is dispersible. It has a “silky” skin feel and is highly writing. It gives great coverage and has an excellent fixation.

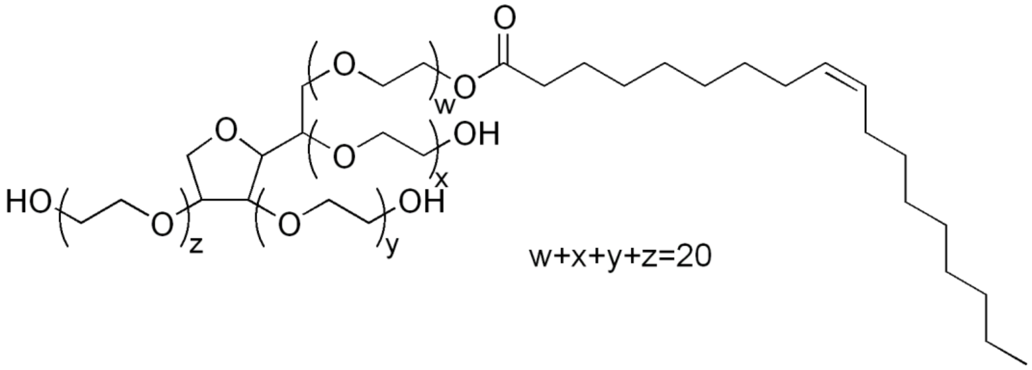
SOLUBILIZERS

- POLYSORBATE 80 – TEGO SMO 80 – SORBITAL T 80 PH

  INCI NAME: Polysorbate 80

  CAS Number: 9005-65-6

  Chemical structure:



Chemical and physical properties: it is a yellowish liquid, with no odor. It is water and alcohol soluble, but it is insoluble in vegetable oils and paraffin oil. HLB value is approximately 15.

Properties: it is a non-ionic surfactant, made by ethoxylation of the dehydrated form of sorbitol: sorbitan. The ethoxylation process adds 80 units of ethylene oxide to the sorbitan molecule. Then esterification with oleic acid occurs, giving the final surfactant.

It is compatible with all other non-ionic emulsifiers and anionic and cationic surfactants.

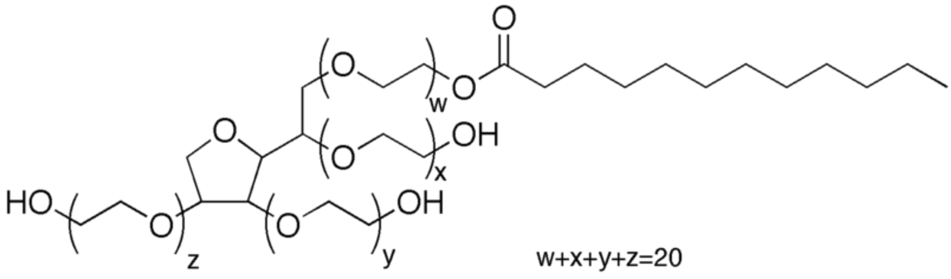
Application: it can be used in shampoos, showers and foam baths due to its mild non-ionic surfactants. It can be utilized as an emulsifier or co-emulsifier for O/W emulsions and as a solubilizer for fatty oils and perfume oils in all aqueous preparations.

- POLYSORBATE 20 – TEGO SML 20 MB

  INCI NAME: Sorbitan monolaurate, ethoxylated/Polysorbate 20

  CAS Number: 9005-64-5

  Chemical structure:



  Chemical and physical properties: it is a dense liquid with no color and has no odor. It is water and cosmetic alcohol soluble, but vegetable oils and paraffin oil insoluble. HLB value is approximately 17.

Properties: it is a non-ionic surfactant, from a vegetable or synthetic source. It is produced by esterification of sorbitol with lauric acid, and subsequent ethoxylation with 20 units of ethylene oxide.

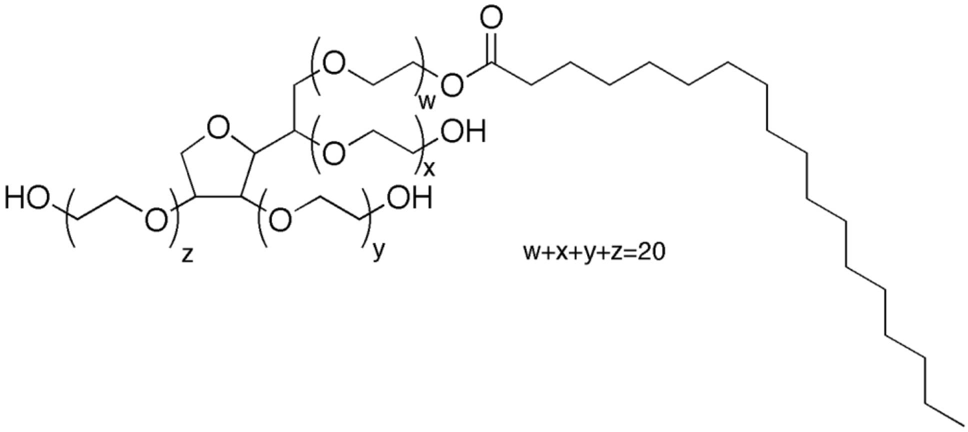
Application: it can be used for hair and skin cleansing products. Acting as a mild surfactant, it can remove impurities from skin and hair without irritation. It can be utilized in O/W emulsions as an emulsifier or co-emulsifier. Due to its action as wetting agent and dispersant, Polysorbate 20 can also be used in cosmetics as foundations, which require cosmetic powder and insoluble pigment dispersion.

- POLYSORBATE 60 – TEGO SMS 60 MB

  INCI NAME: Sorbitan monostearate, ethoxylated/Polysorbate 60

  CAS Number: 9005-67-8

  Chemical structure:



Chemical and physical properties: it is pasty/pourable with no color and no odor. It is water cosmetic alcohol soluble, but vegetable oils and paraffin oil insoluble. HLB value is approximately 15.

Properties: it is a non-ionic surfactant from vegetable or synthetic sources. It is produced by ethoxylation with 60 units of ethylene oxide of sorbitan and subsequent esterification with stearic acid.

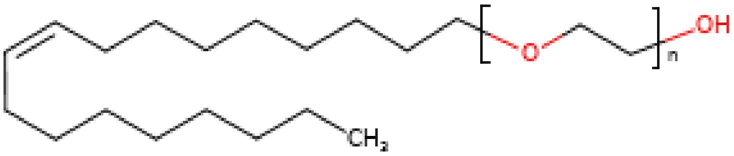
Application: it has application in hair and body care formulas to wash away oils and dirt. It can also solubilize lipophilic substances such as perfumes in water.

- OLETH-20

INCI NAME: Oleth-20

CAS Number: 68920-66-1

Chemical structure:



Chemical and physical properties: it is a clear liquid at 50 °C, while at room temperature is a whiteish solid. When warmed up, Oleth-20 assumes a yellowish color and acquires a kind a characteristic sweetish odor.

Properties: it is an ethoxylated compound of oleyl alcohol with 20 units of ethylene oxide in its structure.

Application: it is used in haircare products, such as shampoos and conditioners, and skincare products. In haircare products, it is widely utilized due to its action as a washing agent and its antistatic property. A particular use of Oleth-20 is in beard gel and shaving gel, stick deodorant and hair coloring.

HUMECTANTS

- GLYCERIN

  INCI NAME: Glycerin

  CAS Number: 56-81-5

  Chemical and physical properties: it is a syrupy, colorless, hygroscopic liquid. It is miscible in water and alcohol but insoluble in oils, fats, and essential oils.

  Properties: it is a triol.

  Application: it is used as a fragrance ingredient, hair conditioning agent, skin conditioning agent, and viscosity decreasing agent [Becker *et al.,* 2019].

- PROPANEDIOL

  INCI NAME: Propanediol

  CAS Number: 57-55-6

  Chemical and physical properties: it is a colorless, odorless liquid.

  Properties: it is a derivative of starch, produced from renewable sources.

  Application: in haircare products, it is applied to increase hydration and can be found in shampoos, conditioners, and styling products. It improves hair detangling and combing while reducing frizz. In skincare, it is used to enhance skin moisturize, also in formulas for oily skin.

- NATURAL BUTYLENE GLYCOL

  INCI NAME: Butylene glycol

  CAS Number: 6290-03-5

  Chemical and physical properties: it is a clear, colorless liquid, with a sweetish characteristic odor.

Properties: it is a diol made from an innovative fermentation process which converts renewable plant sugars into a high-purity butylene glycol, eliminating the need to use fuels as a feedstock. It conforms to ISO 16128 as a natural ingredient with a natural index of 1.

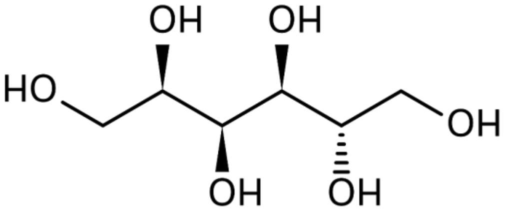
Application: it is used in a variety of personal care and cosmetic applications. It can also be utilized as a solvent and stabilizer for extracts and actives.

- SORBITOL NEOSORB® 70/70 B

  INCI NAME: Sorbitol

  CAS Number: 50-70-4

  Chemical structure:



Chemical and physical properties: it is a syrupy liquid, clear and colorless, and miscible with water.

Properties: it is an aqueous solution of hydrogenated, partly hydrolyzed starch.

Application: it is found in cosmetic products and personal care products, in particular in lotions, gels, toothpastes, and creams. Sorbitol has the peculiar characteristic of potentiation of the preservative system, due to its capability of retaining water.

- DIABOLIK HUMECTANT – Ingredient secreted by the Company.

DISPERSANT – Ingredient secreted by the Company

- DIABOLIK DISPERSANT

  Chemical and physical properties: it is a fine black powder with a sweetish odor. It is highly pigmented and releases a proper quantity of pigment on paper and skin. When dispersed into the water, a brownish halo can be seen.

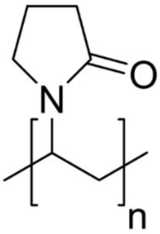
FORM-FILMING AGENTS

- PVP

  INCI NAME: Polyvinylpyrrolidone

  CAS Number: 9003-39-8

  Chemical structure:



  Chemical and physical properties: it is a white to slightly creamy white hygroscopic flakes. It is soluble in water, in methanol and in alcohol.

  Properties: it is a polymer of 1-vinyl-2-pyrrolidone. Its molecular weight can vary based on the length of the polymeric chain.

  Application: PVP is used as a filming agent and stabilizer of emulsions, dispersions, and suspensions. It is also used in gel, mousse, and froth, as well as fixing spray and styling products. It can be found in cosmetics such as eyeliner and mascara due to PVP's peculiarity of maintaining insoluble solids in water suspension and avoiding separation in emulsion.

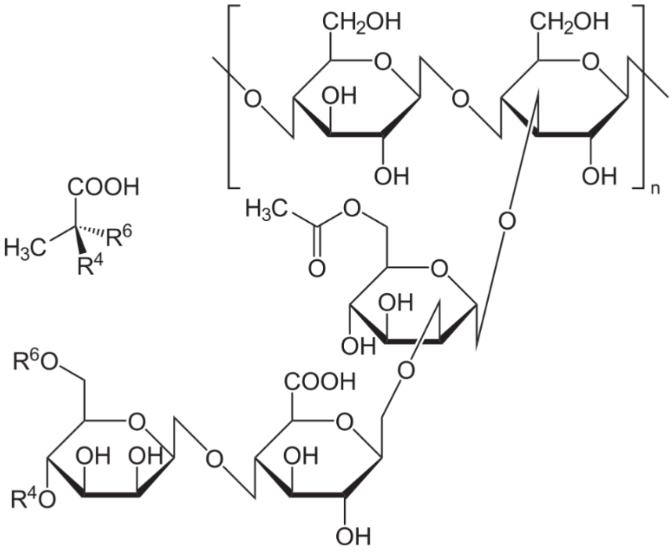
THICKENING AGENTS

- XANTHAN GUM

  INCI NAME: Xanthan gum

  CAS Number: 1138-66-2

  Chemical structure:



  Chemical and physical properties: it is a fine, yellowish powder with a characteristic odor. It is water soluble and soluble in polar solvents.

  Properties: It is a natural gelling agent obtained from the bacterial fermentation of vegetable-based raw materials.

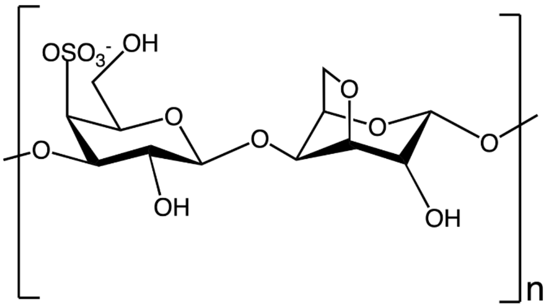
  Application: it is used as a thickener, emollient, surfactant, and stabilizer.

- CARRAGEENAN κ

  INCI NAME: Carrageenan

  CAS Number: 9000-07-1

  Chemical structure:



Chemical and physical properties: it is a fine ambered powder, without odor. It is soluble in water and polar solvents.

Properties: it is a κ carrageenan, potassium salt.

Application: it is used to formulate emulsion and shower gel.

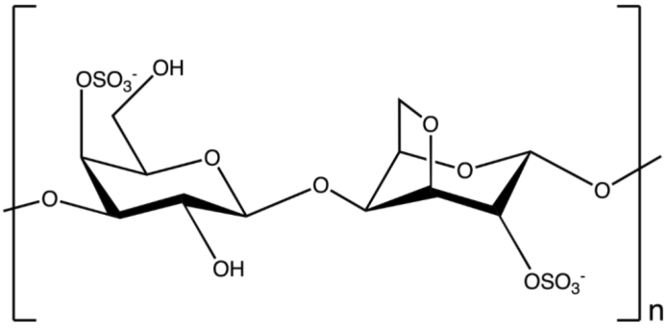
Certification: ECOCERT®

- CARRAGEENAN ι

INCI NAME: Carrageenan

CAS Number: 9000-07-1

Chemical structure:



Chemical and physical properties: it is a fine, odorless, whiteish powder. it is soluble in water and polar solvents.

Properties: it is an ι carrageenan, sodic salt.

Application: it can be used in haircare to formulate shampoos, in body care to formulate soaps, body washes and toothpastes.

Certification: ECOCERT®

INSTRUMENTS

- **ANALYTICAL BALANCE:** KERN PLJ 2000-3°.
- **TECHNICAL BALANCE:** SARTORIUS LP620S**.**
- **HOMOGENIZER** IKA® ULTRA-TURRAX® T25 digital, S25 N.

METHODS

Different trials were conducted to evaluate the dispersion composition that performed most effectively for its intended purpose: dispersing 25% of carbon black, ensuring a stable and homogenous tattoo ink mixture. The experimental approach was non-standardized, as the formula to achieve the optimal dispersion of tattoo inks continuously varied.

In each trial, concentrations and types of raw materials changed, with the only constants being the pigment and the vehicle.

Specifically, the pigment remained stable in composition and concentration, which was set at 25%.

Even though the method cannot be precisely standardized, there is a common thread among all the experimental trials.

Usually, a water-glycerin mixture was prepared, carbon black was added, and then a solubilizer, a surfactant, or a humectant was added. In some cases, even their combination was used to improve dispersion.

1. Solubilizers

During the various tests, different kinds of polysorbate were used for the solubilization of carbon black (Table 3).

In particular, Polysorbate 80, Polysorbate 60, and Polysorbate 20 were used. Only one trial was performed with SORBITAL T 80 PH, but after that, it was chosen to switch to TEGO SMO 80 to utilize polysorbates from the same producer.

Table 3 - Carbon black dispersion with solubilizers.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
SORBITAL T 80 PH	POLYSORBATE 80	1 – 20	Solubilizer
OLETH-20	OLETH-20	6 – 9	
TEGO SMO 80	POLYSORBATE 80	20	
TEGO SMS 60 MB	POLYSORBATE 60		
TEGO SML 20 MB	POLYSORBATE 20		
GLYCERIN	GLYCERIN	4 – 9	Humectant
WATER	AQUA	qb 100	Solvent

1.1. Polysorbate 80

Some tests were made to evaluate the impact of Polysorbate 80 on pigment dispersion, focusing on system stability and ink performance.

Various trials were conducted with Polysorbate 80 of two different brands: SORBITAL T 80 PH and TEGO SMO 80.

In the different trials with the two kinds of polysorbates, the SORBITAL T 80 PH concentration was used in a range between 1% and 20%, while TEGO SMO 80 remained stable at 20%.

1.2. Polysorbate 60 and Polysorbate 20

Polysorbate 60 and Polysorbate 20, another solubilizer from the polysorbate family, characterized by different states of polyoxyethylation compared to Polysorbate 80, were chosen, specifically, TEGO SMS 60 MB (INCI NAME Polysorbate 60) and TEGO SML 20 MB (INCI NAME Polysorbate 20).

Carbon black and TEGO SMS 60 MB were incorporated in known volumes in a water-glycerin solution to evaluate rheology's modifications.

Known quantities of carbon black were dispersed into a water-glycerin mixture, and then TEGO SML 20 MB was added while stirring.

1.3. Oleth-20

Oleth-20 is an ethoxylated compound that was introduced as a substitute for polysorbate because of its effective solubilizing and dispersing properties.

Two different trials were conducted, varying the concentrations of glycerin and Oleth-20 varied, covering a range from 5% to 9% for glycerin and 6% to 9% for Oleth-20.

2. Humectant and wetting agent

Other tests were conducted by utilizing a combination of Polysorbate 20 as a solubilizer and a wetting agent.

Propanediol, butylene glycol, and sorbitol were used as wetting agents (Table 4).

Table 4 - Carbon black dispersion in TEGO SML 20 MB and wetting agents.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6 – 20	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
NATURAL BUTYLENE GLYCOLE	BUTYLENE GLYCOL		
NEOSORB	SORBITOL	4 – 13	
GLYCERIN	GLYCERIN	1 – 9	Humectant
WATER	AQUA	qb 100	Solvent

2.1. Propanediol

Propanediol was added to the water-glycerin mixture, in which it was already added the solubilizer, to obtain a synergism between the solubilizer and surfactant and therefore improve dispersion.

Carbon black, TEGO SML 20 MB in different concentrations (from 10% to 20%) and propanediol were added to the water-glycerin mixture. The wetting agent was used at various concentrations, from 1% to 4%.

2.2. Butylene glycol

For the same reason described above, butylene glycol was added to the formula in substitution of propanediol.

The water-glycerin mixture was prepared, and then butylene glycol was added. Then, carbon black and TEGO SML 20 MB were added by alternating carbon black powder to the solubilizer until the proper concentration was reached.

2.3. Sorbitol

The water-glycerin mixture, after the addition of carbon black and TEGO SML 20 MB, set at 6%, NEOSORB 70/70 B (INCI NAME Sorbitol) was added in variable concentrations, from 4% to 13%.

In these trials, even the concentration of glycerin was changed, with a range of 5% – 9% use.

2.4. Diabolik

To the water-glycerin mixture, after carbon black was introduced, Diabolik Humectant was added in a range from 5% to 10% (Table 5).

Table 5 - Carbon black dispersion with Diabolik humectant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	5 – 10	Humectant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

3. Diabolik dispersant

The dispersant was added to the carbon black, and then water, glycerin, and solubilizer were added.

One trial was carried out using only a dispersant called Diabolik dispersant. The other trials were performed using a mix of a dispersant (Diabolik Dispersant) and a humectant called Diabolik Humectant (Table 6).

Table 6 - Carbon black dispersion with Diabolik Humectant and Diabolik Dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25.0	Colorant
DIABOLIK HUMECTANT*	---	0.7 – 4.0	Humectant
DIABOLIK DISPERSANT**	---	3.0 – 6.0	Dispersant
GLYCERIN	GLYCERIN	0.0 – 5.0	Humectant
WATER	AQUA	qb 100.0	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

Several tests were conducted using various concentrations of dispersant and humectant. Different glycerin concentrations were also used, and its possible removal was also evaluated.

4. Film-forming agents

The carbon black dispersion in which PVP (INCI NAME Polyvinylpyrrolidone) was introduced was prepared by another method.

Firstly, PVP was solubilized in a glycerin solution. At the same time, a dispersion containing water, carbon black, and TEGO SML 20 MB was made and then mixed.

5. Rheology modifiers

The method of making the dispersion was slightly modified to execute other tests in which rheology modifiers were used. First, a powder mix of carbon black and xanthan gum or carrageenan was made. Then, water and TEGO SMO 80 were added in variable concentrations from 10% to 13%.

The rheology modifiers used were xanthan gum, carrageenan κ and carrageenan ι (Table 7).

Table 7 - Carbon black dispersion in TEGO SMO 80 and suspending agents and stabilizers.

TRADE NAME	INCI NAME	QUANTITY (%)	FUCNTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMO 80	POLYSORBATE 80	10 – 13	Solubilizer
XANTHAN GUM	XANTHAN GUM	1	Suspending agent and stabilizer
CARRAGENAN κ	CARRAGENAN		
CARRAGENAN ι	CARRAGENAN		
GLYCERIN	GLYCERIN	2	Humectant
WATER	AQUA	qb 100	Solvent

RESULTS

1. Solubilizers

1.1. Polysorbate 80

The pigment incorporation in the water-glycerin mixture formed a thick, viscous paste because the particles tended to aggregate. Hence, a first trial was conducted with a solubilizer, SORBITAL T 80 PH (INCI NAME: Polysorbate 80): it was introduced to aid dispersion only when the paste was obtained (Table 8).

Table 8 - Carbon black dispersion in SORBITAL T 80 PH.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
SORBITAL T 80 PH	POLYSORBATE 80	1	Solubilizer
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

The quantity of solubilizer was insufficient; therefore, to obtain a homogeneous and liquid dispersion, the percentage of this solubilizer was doubled (Table 9).

Table 9 - Carbon black dispersion in SORBITAL T 80 PH.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
SORBITAL T 80 PH	POLYSORBATE 80	20	Solubilizer
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

The dispersion shown in Table 8 was examined at the drop test. A drop of the dispersion was released into a becher containing water, and the test revealed a rapid dispersion of the drop into the water. Still the dispersion obtained was not homogeneous, and sedimentation occurred eventually. Stirring was necessary to get a black, intense solution.

The pigment did not migrate on paper. It settled in the center of the drop released on the paper and showed a concavity on top.

After the tests were completed, the formulation was recalibrated, and SORBITAL T 80 PH increased to 20%. Other concentrations remained stable.

After the reformulation, the empirical tests were made again, but no differences were observed.

In the subsequent trial, SORBITAL T 80 PH was replaced with TEGO SMO 80 (INCI NAME: Polysorbate 80) as shown in Table 10.

Table 10 - Carbon black dispersion in TEGO SMO 80.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMO 80	POLYSORBATE 80	20	Solubilizer
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

1.2 g of carbon black were added to a water-glycerin mixture, causing a variation in consistency. The formed tick paste returned to a liquid state by adding 0.3 g of TEGO SMO 80. The subsequent addition of 0.7 g of carbon black remade a thick paste, but by adding 0.4 g of TEGO SMO 80, the effect was neutralized. Then again, adding 0.5 g of carbon black changed the rheology of the system. 0.8 g of TEGO SMO 80 was required to bring the dispersion back to the liquid state. In the end, the last addition of 0.1 g of carbon black did not affect the state, which remained liquid by the final addition of 0.5 g of TEGO SMO 80.

The drop test showed that the drop reached complete dispersion after four hours, but sediment was formed on the bottom of the becher.

On paper and skin, there was no migration (Figure 14).

Figure 14 - Spot of carbon black dispersion with TEGO SMO 80.

1.2. Polysorbate 60

After some tests with Polysorbate 80, another polysorbate was chosen (Table 11).

Table 11 - Carbon black dispersion in TEGO SMS 60 MB.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMS 60 MB	POLYSORBATE 60	20	Solubilizer
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

The initial addition of 1.1 g of carbon black to the water-glycerin mixture formed a thick paste that required neutralizing 0.2 g of TEGO SMS 60 MB (INCI NAME: Polysorbate 60). Adding 0.4 g of carbon black changed the rheology again, requiring another 0.3 g of TEGO SMS 60 MB. After that, the ulterior addition of 0.7 g of carbon black formed a thick black paste and 0.7 g of TEGO SMS 60 MB were required to neutralize the system again. Eventually, the last addition of 0.2 g of carbon black produced consistency variations that remained unmodified even after adding 0.8 g of TEGO SMS 60 MB.

The drop test results were promising. Initially, the dispersion drop stayed on top of the water. After that, it dispersed quickly, but stirring was required to obtain apparent homogeneity.

On paper, it was impossible to evaluate whether the product migrates (Figure 15). So, it was tested even on the skin, and no migration was observed.

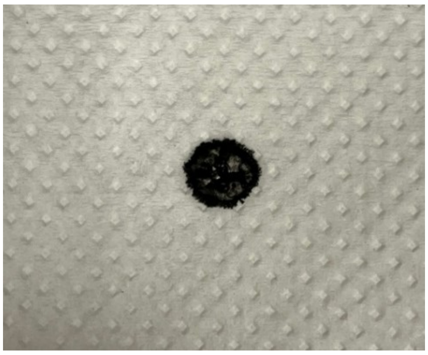


Figure 15 - Spot of carbon black dispersion with TEGO SMS 60 MB.

1.3. Polysorbate 20

Into the next trials, as shown in Table 12, the Polysorbate 60 was substituted with Polysorbate 20.

Table 12 - Carbon black dispersion in TEGO SML 20 MB.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	20	Solubilizer
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

The initial addition of 1.3 g of carbon black to the water-glycerin mixture caused a thick paste to be formed. It has been balanced by adding 0.3 g of TEGO SML 20 MB, repristinating the original system fluidity.

Viscosity increased when another 0.3 g of carbon black was added to the solution, requiring 0.2 g of TEGO SML 20 MB to rebalance the system.

0.5 g of carbon black was added, forming a thick paste. To return it to a liquid state, 0.3 g of TEGO SML 20 MB was added.

The last addition of 0.4 g of carbon black did not affect the system, which remained fluid even after introducing 1.2 g of TEGO SML 20 MB.

The drop test results seemed promising. The drop looked highly dispersible, and the solution, at first grey, became intensely black after stirring. It has sediment at the bottom of the becher and did not show migration on paper and skin (Figure 16).

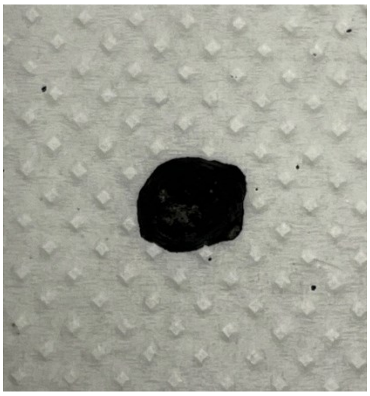


Figure 16 - Spot of carbon black dispersion with TEGO SML 20 MB.

1.4. Oleth-20

In the subsequent trials, Oleth-20 was used instead of polysorbate (Tables 13 and 14).

Table 13 - Carbon black dispersion in Oleth-20.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
OLETH-20	OLETH-20	6	Solubilizer
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

The paper test results were not satisfactory, because there was a minimum migration and a dome-like defect (Figure 17).

Figure 17 - Spot of carbon black dispersion with 6% of Oleth-20.

With the same solubilizer, another trial was made by increasing the percentage of glycerin and Oleth-20, as can be seen in Table 14.

Table 14 - Carbon black dispersion in Oleth-20.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
OLETH-20	OLETH-20	9	Solubilizer
GLYCERIN	GLYCERIN	9	Humectant
WATER	AQUA	qb 100	Solvent

The paper results seemed better than the previous trial. A slight migration occurred, but the swelling mainly stayed the same, even though the dimension of the dome-like formation appeared reduced (Figure 18).

Figure 18 - Spot of carbon black dispersion with 9% of Oleth-20.

2. Humectant and Wetting Agents

2.1. Propanediol

The last trials have shown that the best migration obtained was with TEGO SML 20 MB. So, it was reutilized but in cooperation with a wetting agent (Table 15).

The one chosen was propanediol (INCI NAME: Propanediol) for which every preliminary analysis was executed.

Table 15 - Carbon black dispersion in TEGO SML 20 MB and propanediol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	20	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

Water, glycerin, and propanediol were added sequentially to obtain a watery-like solution. After that, 1.1 g of carbon black was added, and a thick paste was formed. 0.3 g of TEGO SML 20 MB restored the system's fluidity.

0.3 g of TEGO SML 20 MB was necessary to neutralize 0.8 g of carbon black, which caused an increase in viscosity. Then, after having added 0.2 g of carbon black, another 0.7 g of TEGO SML 20 MB was required to obtain a liquid dispersion. The incrementation of 0.2 g of carbon black required 0.5 g of TEGO SML 20 MB to stay fluid. Eventually, the last addition was made of 0.2 g of carbon black and 0.2 g of TEGO SML 20 MB, and nothing changed in the system's rheology.

The drop test showed rapid sedimentation and only slight dispersion after that. Stirring was required to obtain homogeneous dispersions.

On paper, the product seemed capable of a slight migration (Figure 19).

Figure 19 - Spot of carbon black dispersion with 3% of propanediol.

It seemed necessary to reduce glycerin because it synergized with propanediol and TEGO SML 20 MB. This synergism can cause the pigment to be immobilized.

So, concentrations were modified, as shown in Table 16.

Table 16 - Carbon black dispersion in TEGO SML 20 MB and propanediol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	20	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
GLYCERIN	GLYCERIN	2	Humectant
WATER	AQUA	qb 100	solvent

Initially, a water-glycerin mixture was prepared. Then, propanediol, carbon black and TEGO SML 20 MB were added in known quantities.

The initial addition of 1.2 g of carbon black led to a change in viscosity, which was neutralized by adding 0.3 g of TEGO SML 20 MB. After that, a new viscosity change occurred when another 0.5 g of carbon black was added, requiring another 0.2 g of TEGO SML 20 MB to restore the system fluidity. 0.3 g of carbon black had a modified consistency again, making it necessary to add 0.2 g of TEGO SML 20 MB. A subsequent increase of 0.3 g of carbon black modified the system fluidity, making it thick again. The fluidity was restored by adding 0.2 g of TEGO SML 20 MB.

The final addition of 0.2 g of carbon black did not affect the rheology of the dispersion. The process was completed by adding the last 1.1 g of TEGO SML 20 MB

The drop test was a failure. The drop settled at the bottom of the becher and the solution stayed grey.

On the paper sheet, a minimal migration was reached.

An assessment study about the most effective percentage of propanediol was conducted, and the results showed that the concentration could be maintained. Instead, the TEGO SML 20 MB concentration was reduced, as displayed in Table 17.

Table 17 – Carbon black dispersion in TEGO SML 20 MB and propanediol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	10	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
GLYCERIN	GLYCERIN	2	Humectant
WATER	AQUA	qb 100	Solvent

Glycerin, propanediol, carbon black, and TEGO SML 20 MB were added to the distilled water.

The initial addition of 1.2 g of carbon black changed the system rheology, making the dispersion a thick paste. Adding 0.2 g of TEGO SML 20 MB was necessary to neutralize the carbon black and restore fluidity.

After that, the subsequent addition of 0.5 g of carbon black made the same result as before, neutralized by 0.4 g of TEGO SML 20 MB. The last additions of 0.6 g of carbon black and 0.4 g of TEGO SML 20 MB did not affect the rheology, which stayed fluid all along.

The paper test results regarding migration were satisfactory. The ink effectively migrated; however, a dome-like formation appeared at the drop deposition point.

By trying to improve migration, the glycerin concentration was reduced (Table 18).

Table 18 - Carbon black dispersion in TEGO SML 20 MB and propanediol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	10	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
GLYCERIN	GLYCERIN	1	Humectant
WATER	AQUA	qb 100	Solvent

Firstly, a water-glycerin mixture was made in which propanediol, carbon black, and TEGO SML 20 MB were added.

When the initial 1.4 g of carbon black was added, viscosity increased, as usual. 0.3 g of TEGO SML 20 MB was necessary to modify the rheology and make a fluid dispersion. The subsequent addition of 0.8 g of carbon black caused an incrementation of viscosity, neutralized by 0.2 g of TEGO SML 20 MB.

Fluidity got stable even after the last 0.2 g of carbon black addition and 0.5 g of TEGO SML 20 MB.

The paper's results showed that migration did not change while the dome-like formations stood still.

Figure 20 shows the comparison of the different carbon black dispersion spots obtained from the trials, whose concentrations are shown in Tables 16, 17 and 18.

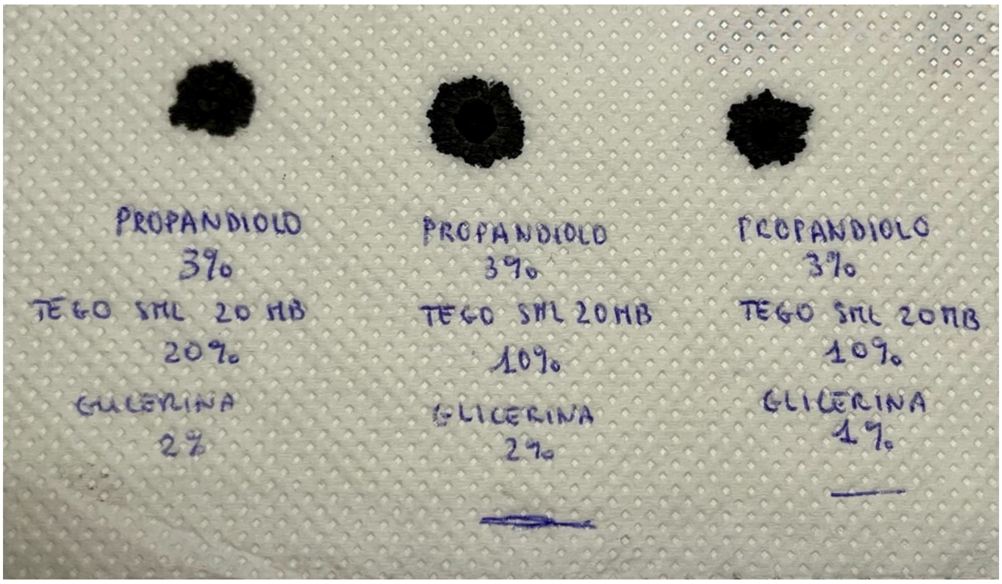


Figure 20 - Spots of carbon black dispersion with propanediol fixed at 3%, with different concentrations of TEGO SML 20 MB and glycerin. .

2.2. Butylene Glycol

Since the bulging effect was not reduced, it seemed necessary to change the humectant. So, it was chosen Natural Butylene Glycol (INCI NAME: Butylene glycol) used in the concentration shown in Table 19.

Table 19 - Carbon black dispersion in TEGO SML 20 MB and butylene glycol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	20	Solubilizer
NATURAL BUTYLENE GLYCOL	BUTYLENE GLYCOL	3	Wettin agent
GLYCERIN	GLYCERIN	2	Humectant
WATER	AQUA	qb 100	Solvent

The initial addition of 1.1 g of carbon black to the water-glycerin mixture containing butylene glycol increased the viscosity, which was neutralized by 0.3 g of TEGO SML 20 MB. Another increment of 1.1 g of carbon black changed the rheology, which was brought back to the liquid state by adding 0.3 g of TEGO SML 20 MB.

The dispersion stayed fluid even after 0.3 g of carbon black and 1.4 g of TEGO SML 20 MB were added, completing the process.

This drop test was the best one. The drop had excellent dispersion, rapidity, and homogeneity, but sediment occurred.

On paper, the test failed (Figure 21). The drop did not migrate, and a bulge was formed.

Figure 21 - Spot of carbon black dispersion with butylene glycol fixed at 3%.

Two things were noticed about all the trials conducted:

1. The formulation with butylene glycol had the best answer to the drop test but the worst dome-like effect
2. By reducing glycerin, the dome-like effect was also reduced

For these reasons, the subsequent trial used both humectants, propanediol and butylene glycol, as can be seen in Table 20.

Table 20 - Carbon black dispersion in TEGO SML 20 MB, propanediol and butylene glycol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6	Solubilizer
PROPANEDIOL	PROPANEDIOL	3	Wetting agent
NATURAL BUTYLEN GLYCOL	BUTYLENE GLYCOL	1	Wetting agent
GLYCERIN	GLYCERIN	1	Humectant
WATER	AQUA	qb 100	Solvent

First, a water-glycerin mixture was prepared. Then, propanediol and butylene glycol were added. The process stays the same. Known volumes of carbon black were added by alternating with TEGO SML 20 MB.

Firstly, 1.2 g of carbon black was added, increasing the dispersion's viscosity. So, adding 0.1 g of TEGO SML 20 MB was necessary to restore the fluidity.

The subsequent addition of 0.5 g of carbon black caused another change in the rheology, neutralized by 0.3 g of TEGO SML 20 MB. The last addition of 0.8 g of carbon black modified the viscosity again, neutralized by 0.2 g of TEGO SML 20 MB, which completed the process, making the dispersion liquid.

Compared to the other tests, the paper test executed on this trial showed that migration and swelling improved (Figure 22).

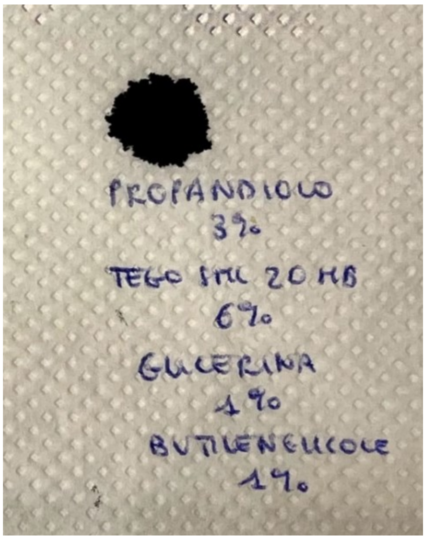


Figure 22 - Spot of carbon black dispersion with both propanediol at 3% and butylene glycol at 1%.

2.3. Sorbitol

To further reduce the dome-like formation, a new wetting agent and humectant were introduced into the formulation (Table 21) by substituting propanediol and butylene glycol.

Table 21 - Carbon black dispersion in TEGO SML 20 MB and sorbitol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6	Solubilizer
NEOSORB 70/70 B	SORBITOL	4	Wetting agent and humectant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Neosorb 70/70 B (INCI NAME: Sorbitol) was added to the hydro-glyceric mixture.

The initial addition of 1.4 g of carbon black caused the dispersion to transition from the liquid state to a thick paste. The effect was neutralized by adding 0.2 g of TEGO SML 20 MB, and the dispersion returned to the liquid state. Then, after di addition of another 0.4 g of carbon black, a semi-solid state was obtained again, which was then liquidized following the addition of 0.1 g of TEGO SML 20 MB.

This alternation between the semi-solid and liquid states was repeated by adding 0.4 g of black carbon and 0.3 g of TEGO SML 20 MB. The dispersion remained liquid even after the last 0.3 g of carbon black addition.

On paper, the migration was acceptable, but no effects on swelling occurred (Figure 23).

Figure 23 - Spot of carbon black dispersion with 4% of sorbitol and 5% of glycerin.

In the next trial, sorbitol and glycerin concentrations were increased (Table 22).

Table 22 - Carbon black dispersion in TEGO SML 20 MB and sorbitol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6	Solubilizer
NEOSORB 70/70 B	SORBITOL	5	Wettin agent and humectant
GLYCERIN	GLYCERIN	9	Humectant
WATER	AQUA	qb 100	Solvent

The procedure stood still. Sorbitol was added to a hydro-glyceric mixture. Then 1.5 g of carbon black was added, and a change in viscosity occurred. 0.3 g of TEGO SML 20 MB neutralized the effect and restored the liquid state.

The usual alternation between states, from semi-solid to liquid, occurred when 0.6 g of carbon black and 0.4 g of TEGO SML 20 MB were added. The liquid state was maintained even after the last addition of 0.4 g of carbon black.

Migration and swelling were better than in the previous trial (Figure 24).

Figure 24 - Spot of carbon black dispersion with 5% of sorbitol and 9% of glycerin.

So, another trial was conducted, in which glycerin concentration was reduced, and sorbitol increased, as Table 23 displays.

Table 23 - Carbon black dispersion in TEGO SML 20 MB and sorbitol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6	Solubilizer
NEOSORB 70/70 B	SORBITOL	13	Wetting agent and humectant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

At first, water and TEGO SML 20 MB were mixed to create a solution. Then, glycerin, sorbitol, and carbon black were added.

The dispersion obtained was homogeneous, and carbon black seemed to have an adequate dispersion. But, after the drop test was made, it was noticed that the dispersion was not satisfactory, appeared grey, and sediment occurred.

On paper, an acceptable migration occurred, even if no differences in swelling were observed than in the other trials (Figure 25).

Figure 25 - Spot of carbon black dispersion with 13% of sorbitol and 5% of glycerin.

With the same productive method, another trial was made by changing glycerin and sorbitol concentrations again. Table 24 shows the new concentrations.

Table 24 - Carbon black dispersion in TEGO SML 20 MB and sorbitol.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	6	Solubilizer
NEOSORB 70/70 B	SORBITOL	9	Wetting agent and humectant
GLYCERIN	GLYCERIN	9	Humectant
WATER	AQUA	qb 100	Solvent

Adding carbon black to the watery solution containing TEGO SML 20 MB, glycerin, and sorbitol created an intense black and homogenous dispersion.

Even if concentrations were different, no changes were observed in the drop test result and the paper test (Figure 26).

Figure 26 - Spot of carbon black dispersion with 9% of sorbitol and 9% of glycerin.

By comparing the different spots on paper, the best migrations were the ones with sorbitol and glycerin, with the concentration fixed to 9% (Figure 27).

The dome-like effect was better in the spot, with glycerin concentration set to 9% and sorbitol set to 5%.

Figure 27 - Spots comparison among carbon black dispersions with different concentrations of sorbitol and glycerin.

3. Film-forming agents

The aim was to improve dispersion, so PVP (INCI NAME: Polyvinylpyrrolidone) was added instead of wetting agents (Table 25).

Table 25 - Carbon black dispersion in TEGO SML 20 MB and PVP.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SML 20 MB	POLYSORBATE 20	3	Solubilizer
PVP	POLYVINYLPYRROLIDONE	1	Film-forming agent
GLYCERIN	GLYCERIN	4	Humectant
WATER	AQUA	qb 100	Solvent

Two mixtures were prepared contemporaneously. PVP was dissolved in glycerin, while a mixture of carbon black, water, and TEGO SML 20 MB was made.

After the two mixtures were ready, they were added together. The product obtained was a thick paste, so dense that it was impossible to take it up from the becher and evaluate it in the empirical trials.

Even after adding 13% propanediol off-formula, the rheology did not change.

4. Rheology modifiers

4.1. Xanthan Gum

Table 26 shows the addition of xanthan gum to control viscosity, texture, and migration.

Table 26 - Carbon black dispersion in TEGO SMO 80 and xanthan gum.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMO 80	POLYSORBATE 80	13	Solubilizer
XANTHAN GUM	XANTHAN GUM	1	Suspending agent and stabilizer
GLYCERIN	GLYCERIN	2	Humectant
WATER	AQUA	qb 100	Solvent

Xanthan Gum was mixed with carbon black. Then the water-glycerin mixture was added, and at least TEGO SMO 80 was introduced.

The paste obtained was so dense that it cannot be evaluated.

4.2. Carrageenan

Another rheology modifier was used to evaluate if the precedent result could be improved.

Carrageenan κ was added in substitution of xanthan gum, as shown in Table 27.

Table 27- Carbon black dispersion in TEGO SMO 80 and carrageenan κ.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMO 80	POLYSORBATE 80	10	Solubilizer
CARRAGEENAN κ	CARRAGEENAN	1	Stabilizer
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Carrageenan κ was mixed with the carbon black, and then water, glycerin, and TEGO SMO 80 were added.

The result obtained was the same as the one obtained with the xanthan gum, and because of that, the paper test and the drop test could not be performed.

Another test was made by changing the carrageenan κ, which was replaced with carrageenan ι. In Table 28, this change is displayed.

Table 28 - Carbon black dispersion in TEGO SMO 80 and carrageenan ι.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
TEGO SMO 80	POLYSORBATE 80	10	Solubilizer
CARRAGEENAN ι	CARRAGEENAN	1	Stabilizer
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Carrageenan ι slightly improved the rheology, but the formulation was dense and thick.

So, the paper test and the drop test could not be conducted.

5. Diabolik family

The Diabolik humectant was added to the basic formulation, as indicated in Table 29.

Table 29 - Carbon black dispersion with Diabolik humectant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	10	Humectant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

A water-glycerin mixture was prepared, and then carbon black was added. Then, Diabolik Humectant was added to the mixture.

On paper, the dome-like formation seemed smaller than usual, but when it dried out, some cuts appeared on it. However, a water halo was made around the drop, and no migration at all occurred (Figure 28).

Figure 28 - Spot of carbon black dispersion with Diabolik humectant fixed at 10%.

In the later trial, the humectant concentration was reduced (Table 30).

Table 30 - Carbon black dispersion with Diabolik humectant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	5	Humectant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

The concentration reduction did not affect the result of the paper test. The dispersion did not migrate at all; a dome-like formation with huge dimensions was formed, and the halo stayed the same as previously (Figure 29).

Figure 29 - Spot of carbon black dispersion with Diabolik humectant fixed at 5%.

During the subsequent trial, TEGO SMO 80 was added to the formula reported in Table 30 (Table 31).

Table 31 - Carbon black dispersion in TEGO SMO 80 with Diabolik humectant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	5	Humectant
TEGO SMO 80	POLYSORBATE 80	5	Solubilizer
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

A water-glycerin mixture was prepared. Carbon black was added, followed by TEGO SMO 80 and Diabolik humectant. On paper, test migration seemed appreciable. No effects occurred on the swelling (Figure 30).

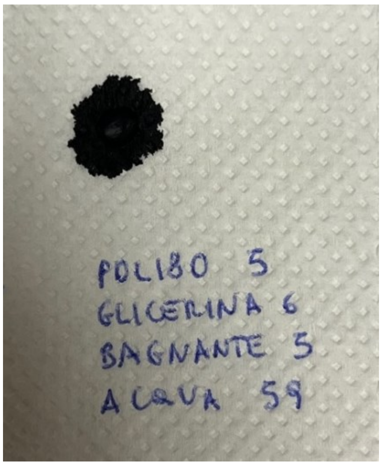


Figure 30 - Spot of carbon black dispersion with Diabolik humectant at 5% and TEGO SMO 80 at 5%.

So, a different test was carried out: the humectant was left over, and the dispersant was added. Table 32 shows the new composition of the formula.

Table 32 - Carbon black dispersion in Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK DISPERSANT**	---	3	Dispersant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note**: black powder with INCI secreted by the company.

The Diabolik dispersant, a fine black powder, was added to the carbon black powder. The mixture was stirred with a glass rod. After that, a water-glycerin mixture, previously made, was added.

The result was homogeneous: on paper, migration was appreciable, and there was a reduction of swelling (Figure 31).

Figure 31 - Spot of carbon black dispersion with Diabolik dispersant at 3%.

In the following trial, humectant and dispersant were put together, as can be seen in Table 33, to improve the dispersion of the pigment into the water-glycerin mixture.

Table 33 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25.0	Colorant
DIABOLIK HUMECTANT*	---	0.7	Humectant
DIABOLIK DISPERSANT**	---	3.0	Dispersant
GLYCERIN	GLYCERIN	5.0	Humectant
WATER	AQUA	qb 100.0	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

The carbon black powder was first mixed with the dispersant, then the humectant, and lastly, the water-glycerin mixture.

Excellent migration was observed on paper, and the dome-like formation was reduced. However, a brownish halo was visible, probably because of the dispersant (Figure 32).

Figure 32 - Spot of carbon black dispersion with Diabolik dispersant at 3% and Diabolik humectant at 0.7%.

With this test, it was noticed that cooperation between Diabolik Humectant and Diabolik dispersant yielded better results.

In trying to reduce the swelling, humectant and dispersant concentrations were increased, as indicated in Table 34.

Table 34 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	1	Humectant
DIABOLIK DISPERSANT**	---	5	Dispersant
GLYCERIN	GLYCERIN	5	Humectant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

Migration on paper can be compared to the previous trial, but there were no effects on the dome-like formation (Figure 33).

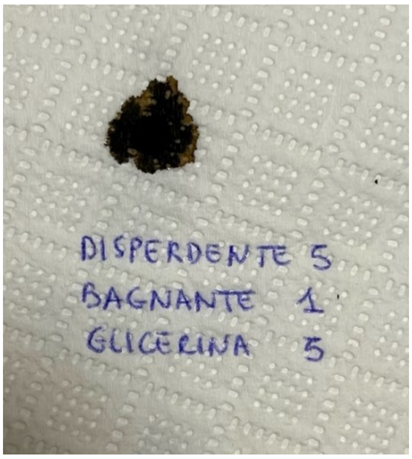


Figure 33 - Spot of carbon black dispersion with both Diabolik humectant and Diabolik dispersant fixed at 5%.

Because of the previous experiments, swelling was evaluated due to glycerin. So, in the subsequent trial, glycerin was removed (Table 35).

Table 35 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	4	Humectant
DIABOLIK DISPERSANT**	---	5	Dispersant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

The paper test result confirmed that glycerin contributed to the swelling. The migration and dome-like formation improved (Figure 34).

Figure 34 - Spot of carbon black dispersion with Diabolik dispersant at 5%, and Diabolik humectant at 4%.

After that, another experiment was made by decreasing the humectant and increasing the dispersant. The concentrations of humectant and dispersant are specified in Table 36.

Table 36 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25.0	Colorant
DIABOLIK HUMECTANT*	---	1.5	Humectant
DIABOLIK DISPERSANT**	---	5.5	Dispersant
WATER	AQUA	qb 100.0	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

These modifications did not improve any aspects of migration and swelling on the paper test (Figure 35).

Figure 35 - Spot of carbon black dispersion with Diabolik dispersant at 5,5% and Diabolik humectant at 1,5%.

Beyond this point, experiments were conducted on a different quantity of the dispersion. Since all trials made until that moment were made on 10 g, which made it impossible to use a mixer, the batch size was increased to 50 g in the following trials.

Table 37 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25	Colorant
DIABOLIK HUMECTANT*	---	3	Humectant
DIABOLIK DISPERSANT**	---	6	Dispersant
WATER	AQUA	qb 100	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

Diabolik dispersant was added to the carbon black powder and mixed with a glass rod. Then Diabolik humectant and water were added. After everything was mixed, homogenization was made with the ULTRA TURRAX (Table 37).

The paper test result was one of the best obtained. Migration was excellent, the swelling was minimal, and there was no halo (Figure 36).

Figure 36 - Spot of carbon black dispersion with Diabolik dispersant at 6% and Diabolik humectant at 3%.

To determine whether the improvement in migration and dome-like effect was due to the concentration ratio between the humectant and the dispersant, an additional experiment was conducted, in which the ratio was maintained at 1:2, but their concentrations were reduced. The new concentrations for Diabolik humectant and dispersant are detailed in Table 38.

Table 38 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	CI 77266	25.0	Colorant
DIABOLIK HUMECTANT*	---	0.8	Humectant
DIABOLIK DISPERSANT**	---	1.6	Dispersant
WATER	AQUA	qb 100.0	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

The paper result was worse than before. Migration was satisfactory, but not as much as the previous one. A brownish halo formed, and the swelling was noticeable (Figure 37).

Figure 37 - Spot of carbon black dispersion with Diabolik dispersant at 1,6% and Diabolik humectant at 0,8%.

Another trial was conducted by maintaining the ratio of 1:2 but reducing concentrations even more. Table 39 shows the new concentrations used in the trial.

Table 39 - Carbon black dispersion with Diabolik humectant and Diabolik dispersant.

TRADE NAME	INCI NAME	QUANTITY (%)	FUNCTION
CARBON BLACK	Ci 77266	25.0	Colorant
DIABOLIK HUMECTANT*	---	0.5	Humectant
DIABOLIK DISPERSANT**	---	1.0	Dispersant
WATER	AQUA	qb 100.0	Solvent

Note*: liquid with INCI secreted by the company.

Note**: black powder with INCI secreted by the company.

The further reduction did not have the desired effect. On paper, migration got worse, a watery halo was formed, and the swelling stayed the same as in the previous trial (Figure 38).

Figure 38 - Spot of carbon black dispersion with Diabolik dispersant at 1,0% and Diabolik humectant at 0,5%.

As a result of the analyses conducted, some observations were made.

Choosing the right dispersant concentration is fundamental to obtaining a performant tattoo ink.

Using insufficient quantities of dispersant leads to instability because of the sedimentation of carbon black particles, while using too much dispersant can cause instability of carbon black dispersion due to the increased interactions between carbon black particles, due to the dispersant itself.

When the dispersant concentration is increased, its efficacy will be reduced. In fact, at low concentrations, the dispersant efficacy increases rapidly as the dispersant concentration rises.

At these low concentrations, the dispersant reduces the agglomeration of carbon black particles without obtaining complete stabilization. While the dispersant concentration rises, its efficacy drops. Further than the hypothetical maximum pick, the efficacy of the dispersant tends to decrease because of its excess, and the carbon black dispersion gets thicker.

The quality of the final carbon black dispersion is strictly dependent on the dispersant concentration. Moreover, another parameter is purely dependent on the dispersant concentration, the dispersion quality.

Dispersion quality increases as dispersant concentration rises. This effect is due to the dispersant capability of separating carbon black particles, which improves the dispersion homogeneity.

When the hypothetical maximum pick is overreached, particles are no longer sufficiently stabilized, and carbon black dispersion quality is reduced, due to the dispersant excess itself and the increased viscosity, which renders the correct distribution of the carbon black particles challenging.

Among the parameters to consider, there is the tendency of carbon black to be dispersible in almost every solubilizer only at low amounts. Inside the ink, there is at least 25% carbon black, and this value can be easily augmented to 30%. At these concentrations, the carbon black concentration increases and is not so readily dispersible.

At low concentrations of carbon black, its particles have sufficient room to be distributed uniformly. At the same time, the concentration of carbon black continues to go up. Carbon black particles are now capable of forming a network without agglomeration.

By then, the dispersion capability starts to decrease while the carbon black concentration continues to go up. Carbon black particles are now near and can interact, forming agglomerates that can compromise the carbon black dispersion.

The particles’ agglomeration is not only possible in the tattoo formulation but in the skin as well.

CONCLUSIONS

In this work, a highly hydrophobic pigment for tattoos, the carbon black Diabolik, was utilized. This pigment is a raw material compliant with REACH restrictions, and achieving its dispersion in a watery solution proves to be challenging. Therefore, various dispersant agents and other ingredients, such as wetting agents and rheology modifiers, were tested.

The main issue encountered was the sedimentation of the carbon black pigment, which negatively affected the ink's stability and usability. Furthermore, migration was another issue, as it is crucial to achieve both effective migration on the skin and to prevent the complete dispersion of the pigment into the dermis.

Although significant progress was made in identifying a potential formula for black ink; however, an optimal formulation has not yet been reached. Further research is needed to develop effective black ink. Other additives, such as solubilizers, surfactants, wetting agents, humectants, and thickening agents should be investigated. Additionally, it is also important to include a preservative in the water-based tattoo ink formula, complying with the current regulations.

Tests on human skin or *ex vivo* can be performed to evaluate properties of tattoo ink, such as fixation, durability, or potential adverse reactions. Moreover, stability studies and microbiological tests must be carried out to assess the shelf life and the performance of the ink over time, as well as to ensure sterility, which is mandatory due to the injection of the tattoo ink formulation under the skin.

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