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The benefits of Fischer-Tropsch waxes in synthetic petroleum jelly

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Synopsis

This article is an introduction and general discussion regarding the use of Fisher-Tropsch wax in petroleum jelly applications. Traditionally, petroleum jelly is prepared from a blend of microwax, paraffin wax and mineral oil that are all derived from crude oil. Sasol Wax has successfully prepared a petroleum jelly based on predominantly to fully synthetic Fisher-Tropsch wax. Sasol Wax was awarded a patent P53898ZP00-29 November 11 for a predominantly to fully synthetic petroleum jelly based on Fisher-Tropsch wax blends. The benefits of Fisher-Tropsch wax discussed in this article include the absence of aromatic compounds and polycyclic aromatic compounds in Fisher-Tropsch wax as well as the sustainable production that is possible with Fisher-Tropsch wax, as opposed to paraffin wax that may be affected by the closure of group I Base Oil plants. This article will be the first in a series of articles from the same authors, and follow-up articles will include solid-state nuclear magnetic resonance and crystallization studies to determine the influence of predominantly synthetic waxes on petroleum jelly network structures compared with more traditional mineral oil-derived petroleum jellies, final product performance and stability of synthetic petroleum jelly used in, for example, personal care lotions or creams. The influence of oxygenated compounds and product safety and rheological properties (including primary skin feel upon application and secondary skin feel after application) of synthetic petroleum jellies compared with traditional mineral oil-derived petroleum jellies are discussed.

Résumé

Cet article est une introduction et une discussion générale sur l'utilisation de la cire Fisher-Tropsch dans les applications de vaseline. Traditionnellement la vaseline est préparée à partir d'un mélange de microcire, la cire de paraffine et l'huile minérale qui sont tous dérivés du pétrole brut. Sasol Wax a préparé avec succès une vaseline sur la base essentiellement, voire entièrement, de la cire synthétique Fisher-Tropsch pour laquel Sasol Wax a obtenu le brevet P53898ZP00-29Nov11 Les avantages de la cire Fisher-Tropsch abordé dans cet article comprend l'absence de composés aromatiques et des composés aromatiques polycycliques, ainsi que le production perenne qui est possible avec de la cire Fischer-Tropsch; par opposition à la cire de paraffine qui peut être affectée par la fermeture des usines d'huiles de base du groupe I. Cet article sera le premier d'une série de articles des mêmes auteurs; les articles suivants comprendront les études de résonance magnétique nucléaire à l'état solide et de la cristallisation afin de déterminer l'influence des cires synthétiques sur les structures du réseau de vaseline, par rapport à celle obtenue plus traditionnellement avec de l'huile minérale ainsi que sur les performances du produit final et la stabilité de la vaseline synthétique utilisée, par exemple dans des lotions ou des crèmes de soins. L'influence des composés oxygénés et la sécurité des produits et des propriétés rhéologiques (y compris primaire de la peau se sentir à la demande et sensation cutanée secondaire après l'application) de la vaseline synthétique par rapport à celle dérivée de l'huile minérale traditionnelle est discutée.

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Introduction

Petroleum jelly has a wide range of applications in cosmetic skin care products such as infant care, colour cosmetics such as lipsticks, hair care products and personal care products, including skin lotions, creams and night creams. Petroleum jelly is also widely used as a pharmaceutical ointment base. Other applications include uses such as leather care products, elastomers, an industrial grease or lubricant, candle wax additives, plasticine, plaster and mould release agents, metal coating, or leather conditioning.

The European Pharmacopeia definition for a white petroleum jelly/petrolatum is a purified and wholly or nearly decolourized mixture of semi-solid hydrocarbons, obtained from petroleum and high-boiling liquid hydrocarbons [1]. The CAS number of white petroleum jelly is 8009-03-8, and the INCI name is petrolatum [2, 3]. References to the petroleum jelly or petrolatum products within the meaning of the European Pharmacopeia are often used without any further specification, especially in older literature. The European Pharmacopeia definition and CAS number 8009-03-8 of petroleum jelly do not define the purity or processing of the product [4]. A wide range of different products may be described by the term petroleum jelly, for example different blended components instead of purified natural petroleum jelly from oil rig precipitation. Blended and natural petroleum jellies are both semi-solid hydrocarbon products from petroleum origin. Petroleum jelly is also known as petrolatum, soft paraffin, paraffin jelly, vaseline, vasoliment, cosmoline, saxolene and stonolene. The U.S. Food and Drug Administration (FDA) recognizes petroleum jelly as an approved over-the-counter skin protector.

As petroleum jelly products are used for human skin applications such as cosmetic or pharmaceutical uses, customers would expect the product to be consistent, and therefore, the identification of the products is required. Several governing bodies exist to govern pharmaceutical products, including petroleum jellies, such as the

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European Pharmacopoeia (5; 01/2005:1799), French Ph. (X), German Ph. (DAB 10), British Ph. (BP 2001) and American Ph. (USP XXIV). Cosmetics regulating bodies such as Colipa (European cosmetics association) also exist to regulate cosmetic products. To commercialize a product in a specific region, a petroleum jelly should meet the requirements of the relevant pharmacopeia or cosmetic association.

With this article, the advantages of using synthetic waxes derived from the Fischer-Tropsch process in petroleum jelly products are discussed including the influence of limited future availability of slack waxes and the specific chemical composition of synthetic waxes. A predominantly synthetic petroleum jelly sample was produced and analysed.

Historical overview

Petroleum jelly was originally promoted as a topical ointment for its healing properties. Petroleum jelly was discovered in 1859 in oil rigs in the United States. Workers used the solid gel material forming on the rigs on cuts and burns they obtained on their skin while working. The oil rig workers found this gel-like product hastened healing. Robert Augustus Chesebrough obtained and investigated this material and refined and purified it. He trade-named the purified jelly as vaseline [5]. In 1875, he founded the Chesebrough Manufacturing Company that in 1955 became Chesebrough-Ponds, a leading manufacturer of personal care products in the United States. R.A. Chesebrough patented the process of making petroleum jelly (U.S. Patent 127 568) in 1872. This product that was 'mined' from oil rigs and purified is often referred to as natural petrolatum.

As Pharmacopoeias only dictate the properties of petroleum jellies and not the origin or method of production, the term petroleum jelly is applied not only to the natural petroleum jellies that are rarely used today, but primarily to mixtures of solid petrolatum (paraffin wax and microwaxes) and liquid paraffinic hydrocarbons as well as 'synthetic petroleum jellies' that comply with the requirements of the Pharmacopoeias. Today, petroleum jellies are predominantly prepared from a blend of microwax, paraffin wax and mineral oil [6–9]. All of these components are derived from crude oil. These types of blended petroleum jellies are referred to as artificial petroleum jellies.

Four main different types of petroleum jellies exist, namely [6–10]:

- Natural petroleum jelly/petrolatum that is a mixture of semisolid hydrocarbons purified from petroleum or crude oil,
- Artificial petroleum jelly/petrolatum that is a blend of natural hydrocarbon waxes (obtained and purified from crude oil) with refined mineral oils,
- Gatch petrolatum is a mixture of by-products of petroleum distillates with paraffin wax and
- Synthetic petroleum jelly contains synthetic hydrocarbons obtained by the Fischer-Tropsch process.

A limited amount of petroleum jellies also contain natural waxes such as Beeswax.

Future availability of Group I Base Oils

Group I Base Oils are obtained from solvent-refining processes, Groups II and III Base Oils are obtained from hydro-processing and refining of crude oil, and Group IV type Base Oils are obtained from a chemical reaction process. Slack waxes are a by-product of the Group I Base Oil production process. Slack waxes are further refined and purified to paraffin waxes that are used in petroleum jelly production. The production process of Groups II–IV Base Oils does not result in slack wax as a by-product or result in lower slack wax volumes than from Group I refineries. The global demand for Group I oils is falling (Fig. 1) as the demand for higher-quality Base Oils (Groups II–IV) increases [11].

Owing to the declining production of slack wax, synthetic wax from Fischer-Tropsch process will become increasingly significant. The petroleum jelly market that utilizes lower-melting slack wax refined to paraffin wax will also be affected.

A wax expansion project is currently ongoing at Sasol Wax (South Africa) to increase the current wax production capability by 60% to more than 10 Kilotons per annum of lower-melting medium wax cuts by 2013. The cost of the project is in the region of ca. €800 Million. The first civil work started early January 2010. This will increase the availability of lower-melting medium wax required to fulfil increasing global demand and therefore also creates the opportunity for petroleum jelly manufacturers to substitute failing slack wax availability with synthetic wax.

Polynuclear aromatic hydrocarbons in crude oil-derived products require careful monitoring

Mineral oil-based petroleum jellies are produced from the heaviest mineral oil refinery fraction or vacuum residue. The high-boiling, carcinogenic components of mineral oil, known as polynuclear aromatic hydrocarbons (PAH), including asphaltenes and polyaromatic hydrocarbons (Fig. 2A–I), tend to be concentrated in this fraction.

Clean-up steps are required to ensure that these compounds are not present in petroleum jelly products. These steps include, for example, propane de-asphalting, hydrogenation, solvent de-waxing, fixed-bed adsorption (bauxite, clay and activated carbon) and sulphuric acid treatment. In relation to the latest technology developments, clay treatment may not be necessary because of very effective hydrogenation units used for the purification of product. Petroleum jelly manufacturers using mineral oil-derived products have to ensure that they implement a clean-up step in their production process to remove PAH.

Careful monitoring of petroleum jelly products is required and recommended by cosmetic regulating bodies such as Colipa and pharmaceutical product regulators, such as the European Pharmacopeia to ensure that they do not contain polycyclic aromatic hydrocarbons. Monitoring steps include measuring UV absorbance maxima for the petroleum jelly as described in 21 CFR 172.880.

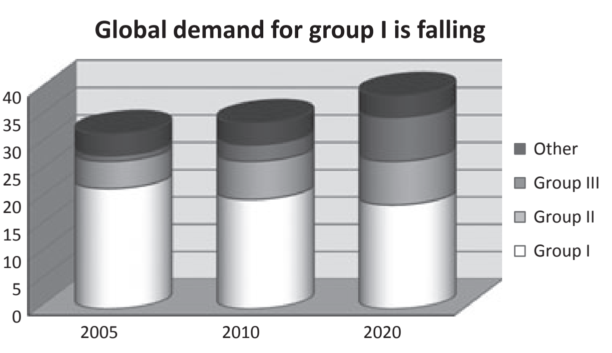

Figure 1 Historical and predicted global demand for Groups I–IV Base Oils.

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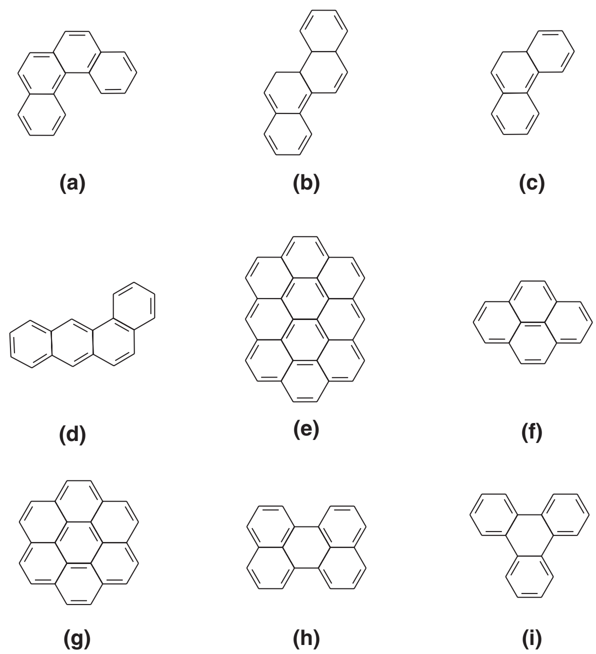

Figure 2 Chemical structure of polynuclear aromatic hydrocarbons.

When the absorbance maxima levels are below the values specified in Table I, the polycyclic aromatic compound levels are below 1 ppm [12].

IR absorbance of polycyclic aromatic hydrocarbons is at the wavelengths shown in Table II. The absence of absorption at these IR wavelengths would also indicate the absence of PAH.

IR analyses of predominantly synthetic petroleum jelly have shown absence of the IR peaks, indicating the presence of polycyclic aromatic hydrocarbons, as well as met the required UV analyses levels as shown in Table I [13].

The classification of petrolatum as a carcinogen on the EU CMR list does not apply if the full crude oil refining history is known and it can be shown that the substance from which it is produced is not a carcinogen. The classification as a carcinogen needs not apply if it can be shown that the substance contains <3% DMSO extract as measured by IP 346. Any petrolatum that passes 21 CFR 172.880 will pass IP 346 by several orders of magnitude.

There is an increased awareness among consumers worldwide regarding the challenges of mineral oil-derived products. Consumer perceptions may not always be an accurate or fair reflection of the truth because petroleum jelly production is strictly controlled and monitored to ascertain that no carcinogenic components are left in

Table I Petrolatum listings in code of federal regulations

21 CFR 172.880 (direct addition to food), 21 CFR 178.3700 (food contact) and 21 CFR 573.720 (animal feed) UV limits	
280–289 nm	0.25 max
290–299 nm	0.20 max
300–359 nm	0.14 max
360–400 nm	0.04 max

Table II Wavelengths of IR absorption for polycyclic aromatic hydrocarbons

Molecule	χ (T)* ($\times 10^{-2}$ cm^{-1} K^{-1})	$\nu_L(0)$* (cm^{-1})	ν_0† (cm^{-1})	$\Delta\nu_{RS}$‡ (cm^{-1})
C—H stretch				
Naphthalene	−2.01§	3074.7	3067.6	7.1
Pyrene	−2.22	3062.9	3066.5	−3.6
Coronene	−3.52	3077.2	3063.7	13.5
Ovalene	−4.90	3082.3	3063.2	19.1
Mean	−3.55	–	–	9.0
C—C stretch				
Coronene 6.2 mm	−4.36	1635.1	1602.7	32.4
Coronene 7.7 μm	−2.38	1325.9	1312.4	13.5
Mean	−3.37	–	–	23.0
C—H in-plane bend				
Pyrene	1.00	1187.9	1188.3	−0.4
Coronene	0.840	1141.5	1140.3	1.2
Mean	0.92	–	–	0.5
C—H out-of-plane bend				
Pyrene	−1.69	847.6	848.3	−0.7
Coronene	−2.30	865.0	864.4	0.6
Mean	−2.00	–	–	0

×(T) increases with increasing PAH size
*Temperature dependent redshifts
†3.4 Mn ab initio frequencies were determined
‡Rounded to nearest 0.5

the commercial products, but it is a fact that a lot of web-based consumer groups have recently been launched to warn against possible impurities in petroleum jelly products. Consumer concerns can be managed either by removing and eliminating carcinogens from petroleum jellies and strict control measures to make sure these carcinogens are not present at unacceptable levels when crude oil-derived material is used, or by introducing a new predominantly or completely synthetic based product, which by its nature does not contain sulphur and aromatic type compounds. Synthetic petroleum jelly products are custom made. Fisher's contact dermatitis recommends synthetic petroleum jelly because a synthetic mixture of hydrocarbons can be more easily controlled [14].

Despite the massive frequency of use of petroleum jellies, only a very few cases of skin irritation or allergic contact dermatitis have been are reported. A total of 14 cases in million fold petroleum jelly daily uses were found in recent literature. Dooms-Goossens reported four cases between 1980 and 1983 of positive allergic reactions to two yellow petroleum jellies [15–18], Lawrence reported one case in 1982 of a positive reaction to white petroleum jelly and one of two yellow petroleum jellies [19], Ayadi reported one case in 1987 of allergic reaction to white petroleum jelly [20], Fisher reported one case in 1981 of allergic reactions to yellow and white petroleum jelly [21], Malten reported one case in 1969 to yellow petroleum jelly [22], Grimalt reported one case in 1978 to yellow and white petroleum jelly [23], Maibach reported one case in 1978 with hyperpigmentation [24], Conti reported one case in 1995 of a positive reaction to four white petroleum jellies [25], Maibach reported one case of allergy to white petroleum jellies in 1999 [26], Kundu reported one case in 2004 [27], and Tam reported one case in 2006 of allergic reactions of damaged skin to white petroleum jelly [28]. In most cases, allergic reactions are to less highly purified 'yellow petroleum jelly'. An allergic reaction to preparations with

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white purified petroleum jelly could also possibly be caused by additives (antioxidants and preservatives) in the products. In the 'Dermatitis' journal, the use of synthetic petroleum jelly is recommended to counter allergic reactions to less-purified petroleum jelly 'if it (synthetic petroleum jelly) can be made to possess qualities and benefits similar to those of natural petroleum jelly' [28].

The Fischer-Tropsch process to produce wax

A low-temperature (200–240°C) Fischer-Tropsch process with either iron or cobalt catalysts is used for the production of high molecular mass linear waxes. Commonly, two types of reactors are used. In top-fed, multitubular reactors (Fig. 3), the wax that is produced trickles down a catalyst bed [28].

In the Sasol (Sasol Ltd., Sasolburg, South Africa) SPBTM slurry bed reactor, the wax that is produced accumulates inside the reactors and needs to be removed. A typical slurry bed reactor is shown in Fig. 4.

The Fischer-Tropsch process occurring in these reactors consists of a syngas (CO) that is hydrogenated in the presence of a catalyst followed by a stepwise growth process on the catalyst surface.

$$CO + H_2 \rightarrow \text{hydrocarbons} + H_2O$$

CH_2 units that are formed by the hydrogenation of CO act as monomers in a stepwise oligomerization process. At each stage, the adsorbed hydrocarbon species can either desorp or continue chain growth by adding another monomer.

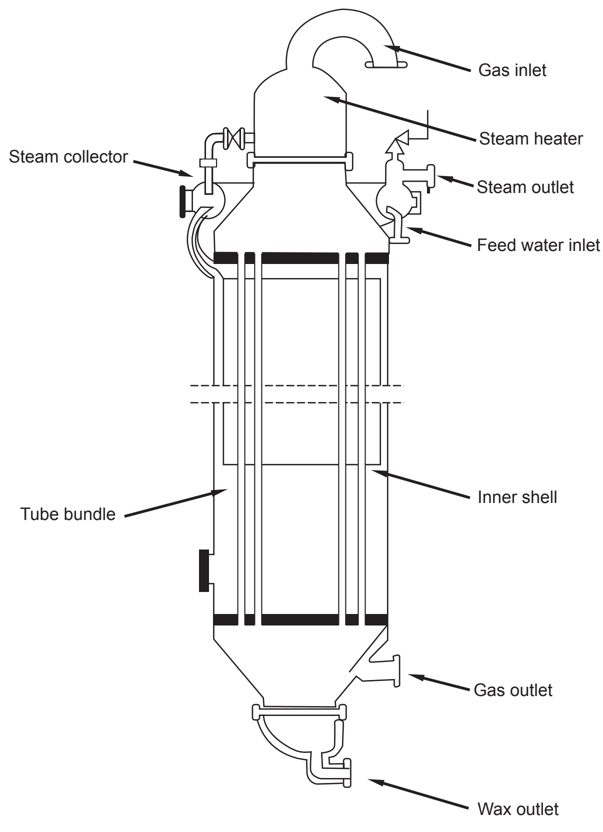

Figure 3 Multitubular fixed-bed Fischer-Tropsch reactor.

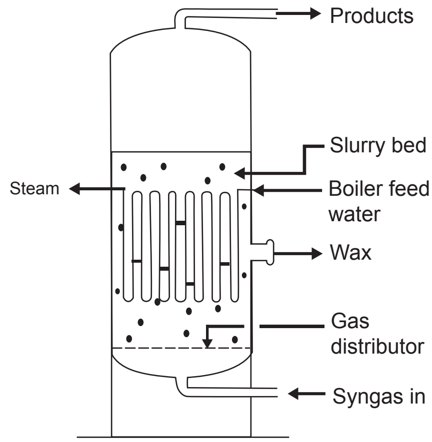

Figure 4 Slurry phase bubbling bed reactor.

Irrespective of operating conditions, the FT process always produces a wide range of mainly paraffins, as well as a small amount of olefins and oxygenated products such as alcohols, aldehydes, acids and ketones. The spread of products is determined by production temperature, feed gas composition, pressure, catalyst type and catalyst promoters. Sulphur and aromatic type compounds are not produced, which is an advantage for the use of synthetic wax in petroleum jelly production. Oxygenated and olefinic compounds may be easily removed by further processing.

Preparation of a predominantly synthetic petroleum jelly

The challenge of producing a predominantly synthetic petroleum jelly lies in the use of synthetic wax that is predominantly made up of straight-chain molecules and a low percentage of molecules with methyl branching, compared with traditional crude oil-derived waxes that contain longer branches and more branched and cyclic molecules. It is expected that crude oil-derived waxes are more suited to building up the required three-dimensional network structure of a petroleum jelly. However, a good quality, predominantly synthetic petroleum jelly was prepared by blending 20% microwax, 55% synthetic medium-melting wax and 25% synthetic paraffin. Preparation of a fully synthetic petroleum jelly is also possible by replacing the microwax component with a synthetic microwax (hydroisomerized synthetic wax). Physical properties of these petroleum jellies are described in Table III.

Two-dimensional GC analyses of the predominantly synthetic petroleum jelly show that no trace amounts of aromatic hydrocarbons are present, in contrast to mineral oil-derived petroleum jellies where these compounds may still be present in ppb levels after refining (Fig. 5).

Fig. 5 shows oxygenate compounds are present in predominantly synthetic petroleum jellies prepared from Fischer-Tropsch waxes.

Identification of the components present in the predominantly synthetic petroleum jelly was performed using GCxGC-TOF(MS) and compared with a traditional petroleum jelly based on mineral oil-derived material containing no synthetic components. Results showed that the predominantly synthetic petroleum jelly mainly (77.1%) contains straight-chain alkane components, 6.7% branched alkanes, as well as some oxygenates, whereas a

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Table III Predominantly and fully synthetic petroleum jelly properties

	Requirement for white Petroleum jelly	Predominantly synthetic PJ	Fully synthetic PJ
Appearance (visual)	White unctuous paste	White unctuous paste	White unctuous paste
Solubility at 25°C (visual)	Water: insoluble Cyclohexane: soluble	Water: insoluble Cyclohexane: soluble	Water: insoluble Cyclohexane: soluble
Drop melting point (ASTM D217)	35–70°C	52.3	62.0
Consistency at 25°C (Eur Pharm)	60–300 mm/10	185	162
UV absorbance	280–289 nm 0.25 max 290–299 nm 0.20 max 300–359 nm 0.14 max 360–400 nm 0.04 max	pass	Pass
Sulphated ash (Eur Pharm)	Pass	Pass	Pass
Alkalinity/Acidity (Eur Pharm)	0.05% max	0.01	0.02

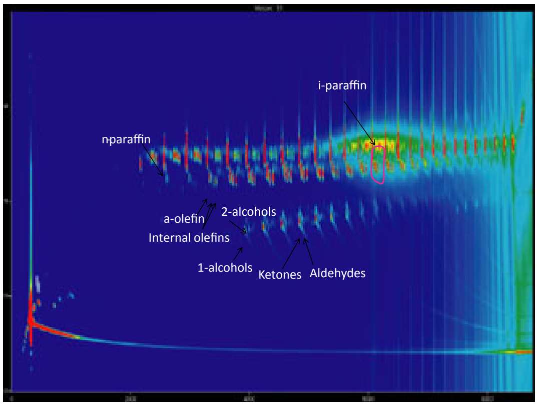

Figure 5 Two-dimensional gas chromatograph of a predominantly synthetic petroleum jelly.

traditional mineral oil-based petroleum jelly predominantly contains branched and cyclic molecules (72%) and only 27% n-alkanes. Predominantly synthetic petroleum jelly contains 0.02% aromatic components compared with 0.84% of the traditional mineral oil-based petroleum jelly.

Conclusions

The availability of slack waxes will decrease in the next decade with the increased global demand for higher-quality Groups II–IV Base Oils. This will mean that less paraffin wax will be produced from slack waxes, and this will impact the availability and cost of petroleum jelly products. Alternative wax sources for the production of petroleum jelly are therefore essential. Synthetic wax obtained from the Sasol SPBTM Fischer-Tropsch process may be successfully used in the production of high-quality petroleum jellies made up of predominantly or exclusively synthetic material. Synthetic wax has the further advantage of not containing sulphur and aromatic impurities. Synthetic wax is custom made, and the manufacturing process can be carefully controlled to determine petroleum jelly properties.

Future work at this laboratory will include the following:

- Solid-state nuclear magnetic resonance and crystallization studies to determine the influence of predominantly synthetic waxes on petroleum jelly network structures compared with more traditional mineral oil-derived petroleum jellies,
- Final product performance and stability of synthetic petroleum jelly used in, for example, personal care lotions or creams,
- The influence of oxygenated compounds,
- Rheological properties of synthetic petroleum jellies compared with traditional mineral oil-derived petroleum jellies.

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