

THE MECHANISM OF HETEROGENEOUS CATALYTIC
HYDROGENATION OF FATTY OILS*Bronisław Drozdowski*Institute of Organic and Food Chemistry and Technology, Technical University
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The hydrogenation is one of the most important chemical reaction in the technology of fats. Although the process of hydrogenation is employed for a long time in the oil industry, a detailed understanding of the mechanism and chemistry of the process is still not reached. This situation is not due to a lack of adequate investigations, as these have been done in a great numbers, but it arises mainly from the extreme complexity of the reacting systems.

The present review is concerned with some aspects of the nature and mechanism of fat hydrogenation, which is, having in mind the industrial process, the classical example of a heterogeneous gas-liquid reaction catalyzed by suspended solids. Therefore, mass and heat transport processes may thus play an important role in reaction kinetics.

Many types of hydrogenation catalysts are known. They consists especially of metals belonging to group VIII of the periodic table. Nickel, platinum and palladium are the most active. A common feature of all these metals is that they have high electron affinities since their outermost or penultimate orbitals are unfilled (Ni — $3d^84s^2$, Pd — $4d^{10}5s^0$, Pt — $5d^96s^1$), which participate in formation of surface bonds with hydrocarbons and hydrogen. Another feature of hydrogenating metals concerns atomic spacings [35, 62]. During hydrogenation of the $-\text{CH}=\text{CH}-$ group, two-point adsorption of the two carbon atoms to the metal is also considered. If the interatomic distance between the metal atoms is about 2,74 Å, the valence angle (two-point adsorption) is very close to that of tetrahedral carbon. These distances for platinum, palladium and nickel are 2.76; 2.74 and 2.47 Å, respectively. Platinum and palladium are more powerful hydrogenation catalysts than nickel. However,

in commercial practice, most of the hydrogenation catalysts are based on nickel.

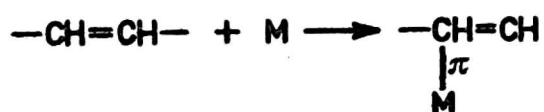
The hydrogenation of fatty oils is the three-phase process. The solid catalyst is suspended in the liquid oil which is brought into the best possible contact with hydrogen.

Catalytic reactions occur on the catalyst surface, and the nature of the latter is therefore of importance. It is generally assumed that the reaction proceeds through the formation of unstable intermediate compounds or adsorption complexes but their exact nature is not definitely known. Chemisorption is recognized by many investigators to be the main type of adsorption but others believe physical one may be the prevalent type. Therefore, the catalyst have the ability to activate both the hydrogen and the unsaturated fatty acid by their adsorption on the active sites of its surface.

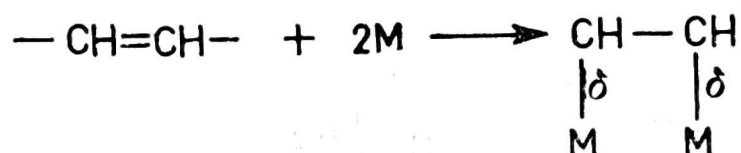
In the more usual low pressure industrial hydrogenation it is believed that the hydrogenating species is the hydrogen atom, although recently attention is given to the participation of molecular hydrogen. Therefore, the activation of the hydrogen molecule by a metal hydrogenation catalyst involves the splitting of the molecule. The hydrogen could be bound by a σ -bond or it is just solved in the metal-lattice. It has been suggested that two forms of chemisorbed hydrogen exist [54], $M-H^-$ for small coverage of the metal surface and $M-H^+$ for high coverages.

During catalytic hydrogenations also olefins are activated by chemisorption through electronic rearrangements [11, 15, 57]. There exist mutual interaction between a double bond and a metallic catalyst. Responsible for the formation of a metal-olefin-bond is the donation of π -electrons of the olefin into empty d -orbitals of the metal and the back donation from filled metal d -orbitals to acceptor π^* -orbitals of the olefin.

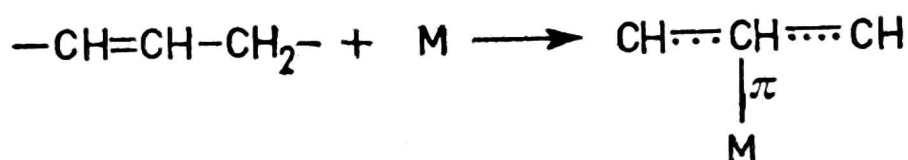
With monoolefins the π -bonded structure involves one π -bond between a single double bond and a single atomic center of the catalyst (M):



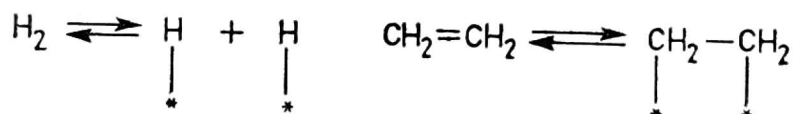
It requires a low energy of activation and no change in hybridization of olefinic carbons from sp^2 to sp^3 . It is assumed that the next step of the mechanism is rearrangement in configuration. The σ -bonded structure, comprising two σ -bonds between saturated (sp^3) carbon and two active sites on the catalyst surface, is formed:



Another possible structure of the chemisorbed olefin is the π -allyl structure [55] which may be formed with olefin containing α -methylenes by splitting of one hydrogen atom from a CH_2 — group adjacent to the double bond:

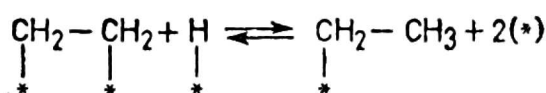


The current concept of the hydrogenation of olefins was advanced by Horiuti and Polanyi [33]. According to this classical mechanism hydrogen and unsaturated molecules are first activated by chemisorption on the active surface of the metal:

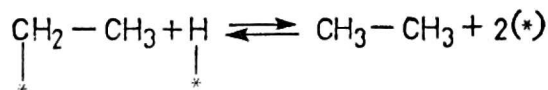


where (*) represents an active surface site.

Hydrogenations passes through an intermediate state, the "half-hydrogenated" state:

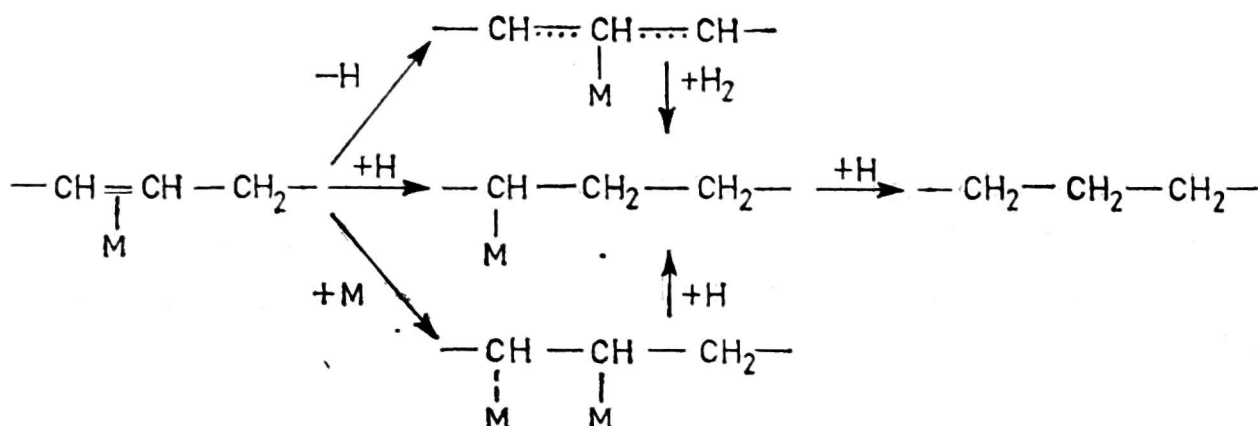


Saturation occurs by reaction of the half-hydrogenated state with another adsorbed hydrogen atom:



The last reaction is effectively irreversible under the usual operating conditions employed in hydrogenation of fatty oils.

Considering the higher olefins, the half-hydrogenated intermediate may be recognised [15, 30] as a σ -monoadsorbed alkyl radical which may be formed in three different ways:

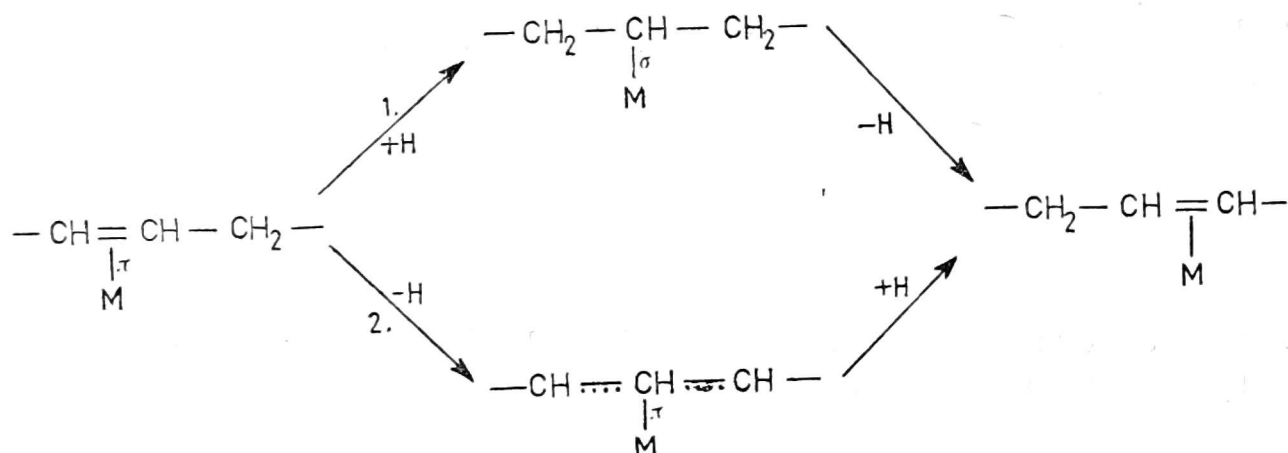


Individual steps in catalytic reaction are essentially reversible. Therefore, the half-hydrogenated intermediate by giving back a hydrogen atom to the catalyst may return to the (adsorbed) alkenic state. This dehydrogenation mechanism introduces the isomerisation reaction — positional and geometrical (cis-trans).

The double bond migration and cis-trans isomerisation may proceed by two different mechanisms [14, 15, 30, 56]:

1) hydrogen addition — elimination through a σ -monoadsorbed intermediate;

2) hydrogen elimination — reincorporation through a π -allyl intermediate.



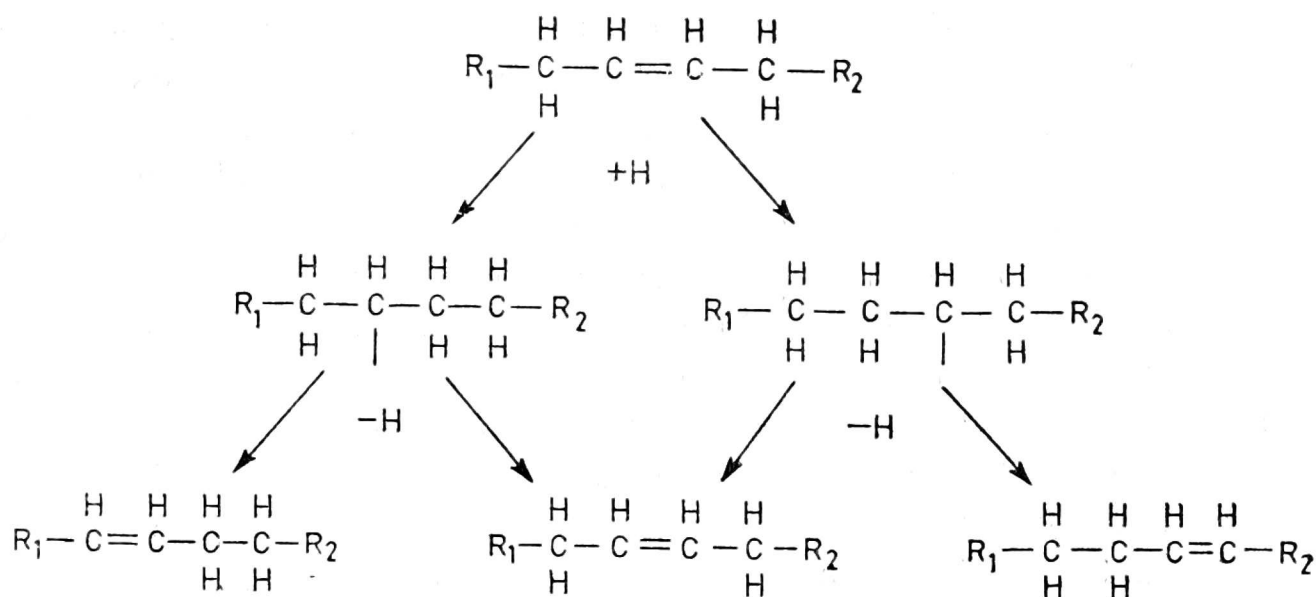
In the σ -monoadsorbed half-hydrogenated state the molecule has the possibility of "free rotation" about the bond between the carbons involved. A rotation of 180° can lead through dehydrogenation to the trans-isomers with or without migration. Splitting of the hydrogen atom taken up originally will reproduce the original double bond (without migration).

The π -allylic mechanism, involving 3 carbon atoms, would give rise to cis-trans rearrangements always accompanied by migration of the double bond, because free rotation is not possible in the adsorbed state.

The existence of the syn- and anti-conformations of the π -allyl intermediates is responsible for yielding cis and trans isomers [14].

The mechanism of isomerisation is governed by the availability of hydrogen on the catalyst surfaces. If the metal surface has a higher coverage of hydrogen atoms, the addition — elimination mechanism would be favoured as the σ -alkyl intermediate is formed by addition of hydrogen contrary to the π -allyl structure which is formed by elimination.

The Horiuti and Polanyi concept of hydrogenation of unsaturated olefins was applied specifically to fat hydrogenation by Blekkingh [13] as partial hydrogenation and partial dehydrogenation steps. This concept of hydrogenation has also been applied by Allen and Kiess [7] to explain the mechanism of positional and geometrical isomerisation:



At the stage of half-hydrogenation of the original double bond the possibility of "free rotation" may lead to cis or trans configuration. The authors indicated that the migration of double bond occurred equally in both sides of the original position and each positional isomer comprised a 1 : 2 equilibrium mixtures of cis- and trans-isomers. Both positional and geometric isomerisation were regarded as simultaneous. Allen and Kiess also indicated that the concentration of hydrogen on the surface of the catalyst influences the rate of isomerisation reaction which was more rapid at low than high concentration. At low hydrogen concentration on the catalyst surface the isomerisation reaction will be favoured over the completion of hydrogenation of half-hydrogenated molecule.

More recently it has been shown that the cis-trans equilibrium is closer to a ratio 1 : 4 [28] and that the cis monoenes to hydrogenate faster than the corresponding trans isomers [58]. The positional isomers

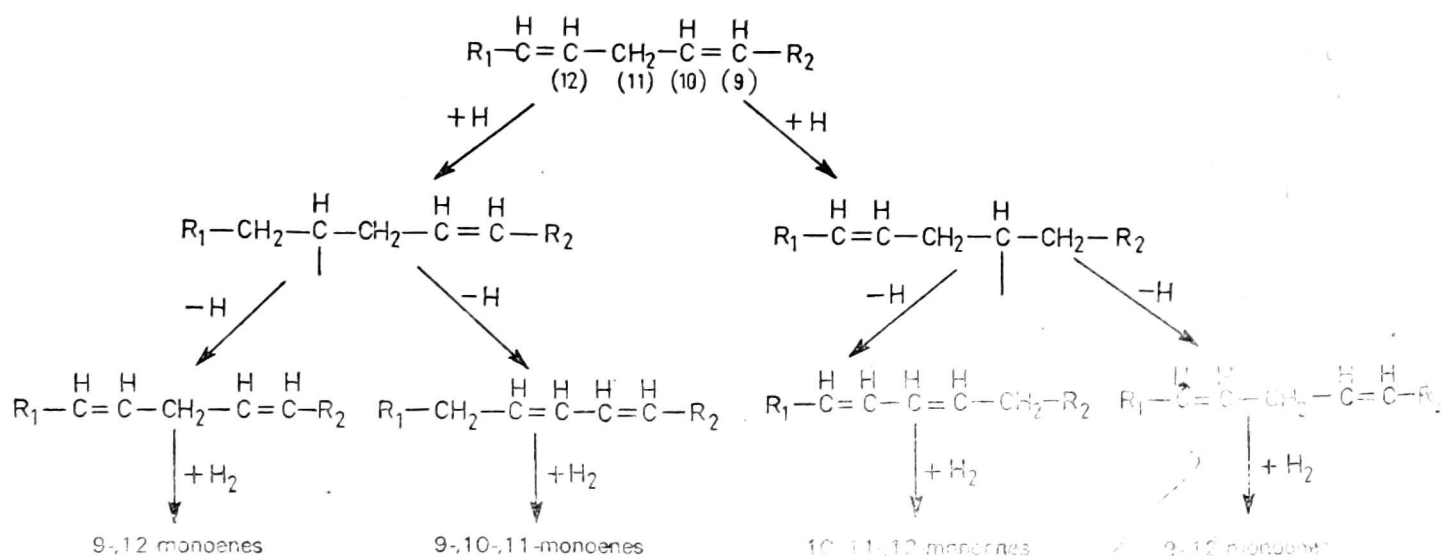
(methyl cis octadec — 6, 9, 12 — enoate) are hydrogenated at the same rate [8].

According to current theories of bonding which were discussed earlier, the monoadsorbed half-hydrogenated intermediate in the half-hydrogenation — dehydrogenation concept, would have a mono σ -bonded structure (a possibility of free rotation), which may be derived either from a π -bonded nonadsorbed intermediate or from a two σ -bonded diadsorbed species [30].

In addition to this isomerisation theory, consistent with the mechanism of Horiuti and Polanyi, the hydrogen elimination — reincorporation process through π -allyl intermediates should be also considered [14, 30, 32, 44, 52, 53]. There are suggestions that both processes are operating simultaneously and that the allylic and half-hydrogenated intermediates are formed at the different sites of the catalyst.

So far this discussion has been limited to molecules with a single double bond. Natural glycerides are composed of fatty acids having either a saturated or an unsaturated character with one or more double bonds. It is known in turn that polyene fatty acids are adsorbed more strongly on the surface of catalyst than fatty acids with one double bond. Therefore, these polyunsaturated acids will tend to monopolise the surface and will go preferentially into reaction [18]. Extensive work has been done with linoleic and linolenic acid. The selective hydrogenation of these two acids over oleic acid has been established.

In most naturally occurring polyunsaturated acids the double bonds are separated by a methylene group. Because of the presence of this active group the conjugation of the double bonds would be expected as intermediate formation [29, 30]. As monoene distribution in the products obtained from linoleate cannot be explained by a simple conjugation Allen and Kiess [9] proposed the half hydrogenation — dehydrogenation mechanism, including conjugation as a major path:

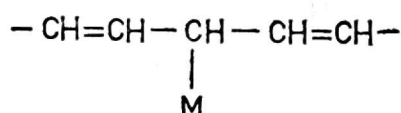


The unrecated double bond would retain its original *cis* configuration but the newly created one is found in practice to be entirely of *trans* configuration. Isolated dienes undergo 1,2-addition of hydrogen to yield 9- and 12-monoenes, whereas the conjugated dienes are hydrogenated by 1,2- and 1,4-addition giving a mixture of 9-, 10-, 11- and 12-monoenes. The authors suggested that under selective conditions of reaction isomerisation would be favoured over hydrogenation and the product obtained from the more reactive conjugated diene structures would predominate. This mechanism however does not explain greater reactivity of linoleate than oleate.

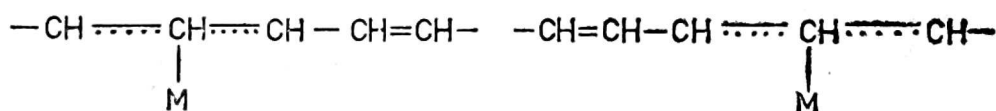
Coenen and Boerma [18, 19] postulated that under hydrogen shortage one double bond of linoleate is first adsorbed similar to adsorption of oleate and after splitting off hydrogen atom from the reactive methylene group the acid is bonded with three adjacent CH- groups. Then the second double bond may be adsorbed leading to a "five-bonded" intermediate. By addition of hydrogen atom to this structure (at either end of 5 CH- groups) it would yield conjugated dienes strongly chemisorbed with surface.

If the hydrogen coverage is high linoleate bonded first with only one double bond may directly be converted into half-hydrogenated structure by taking one hydrogen atom. By taking up a second H-atom this double bond may be reduced completely. Under these conditions the bonding strength to the metal surface by poly- and singly-unsaturated acids will not differ much (low selectivity).

The allylic mechanism has been also postulated [30] under conditions of low hydrogen coverage (selective conditions). Hydrogen abstraction from C-11 of linoleate leads to a σ -bonded diene structure:



which is converted to allylic species:



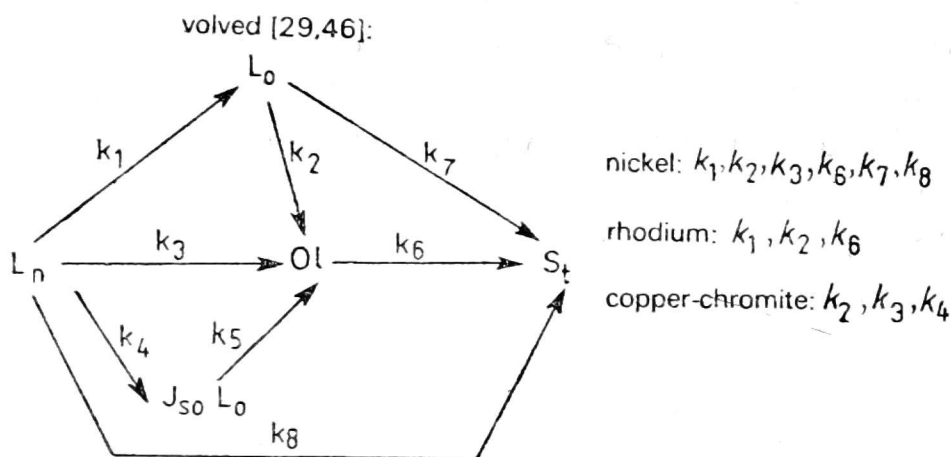
Hydrogen addition yields chemisorbed *cis*, *trans*-conjugated dienes. The 1,2- and 1,4-addition of hydrogen to these dienes will produce the different *cis*- and *trans*-monoenes.

It has been proved [37] that conjugated dienes in the mixture with

isolated ones are preferentially chemisorbed on the catalyst surface.

The methyl linolenate hydrogenation mechanism was studied by many authors. The formation of a partial conjugated (dienetrienes) structures of linolenate is assumed with both half-hydrogenation-dehydrogenation and π -allyl mechanisms. However to explain all the products of linolenate hydrogenation other concepts than partial conjugation must be invoked.

Different metal catalysts have a characteristic effect on the kinetics and the mechanism of catalytic hydrogenation of polyene acids and different kinetic patterns may be involved [29, 46]:



Copper catalysts have an exceptional high selectivity of linolenic acid hydrogenation comparing with all other hydrogenation catalysts. The ratio of the rate constants for hydrogenation of linolenic and linoleic acid is generally between 8 and 15, depending among others on the method of preparation of catalyst [34, 36, 38, 39, 50]. The hydrogenation with copper catalyst proceeds almost exclusively through conjugated intermediate [40-43]. Nonconjugatable polyenic acids and monoenic acids are not reduced with copper catalysts. Since single double bonds are not hydrogenated or isomerised by that catalyst, the hydrogenation of linolenate with wide distribution of monoene products suggests that conjugated dienes undergo migration as a unit. Depending on the hydrogen concentration on the catalyst surface, this migration may occur either through π — or $\pi\sigma$ -diadsorbed half-hydrogenated or π -allyl intermediate [30].

During the hydrogenation of a natural oils not only the most unsaturated acyls are hydrogenated but at the same time all the others which are present in the system may be hydrogenated. The rates at which these reactions occur describe the reaction selectivity. The problem of selectivity is very important not only for the stability of the hydrogenated product but also from the nutritional point of view. The selectivity is defined as the ratio of reactoin rates. According to the

classical definition it is considered as the ratio of hydrogenation rates of linoleic and oleic acids ($S_{2/1}$). When an oil contains higher unsaturated fatty acids such as linolenic acid, the term of selectivity may be defined as the relative rates of reaction for the polyunsaturated groups as compared to the monounsaturated ones. This kind of selectivity is strongly dependent on reaction conditions and can reach values as high as 100.

For certain types of application, especially when copper catalysts are used, the selectivity may be considered as the preferential hydrogenation of fatty acids with more than two double bonds. Copper catalysts show an unusually high selectivity towards linolenate hydrogenation in the liquid phase [34, 37, 38, 39, 50] and relative rates of reaction for the linolenic acid as compared to the linoleic acid ($S_{3/2}$) vary usually between 12-15. For the nickel catalyst $S_{3/2}$ is always rather low (about 2).

If the compositions of both the initial oil (before hydrogenation) and the partially hydrogenated oil are known than the selectivity ratio can be calculated by using a computer [16, 17, 46] or may be estimated graphically [2, 10, 59].

However, fatty oils do not consist of fatty acids, but of triglycerides. It is generally acknowledged that fatty acids in them are not randomly distributed among the individual positions. Considering the steric effect, it may be expected that the catalytic hydrogenation of unsaturated acyls should take place at different rates, depending on the position occupied in triglycerides. The studies in our laboratory on the structure of partially hydrogenated triglycerides have proved that assumption [22]. The unsaturated acyls in the outer positions are more reactive. This phenomenon should be included to the concept of selectivity.

On the basis of mechanistic considerations, carried out previously, it may be formulated that the selectivity as well as isomerization are controlled by the concentrations of reactants at the catalyst surface [3-5, 7, 18, 30, 31, 44, 52]. Therefore, the mass transfer of reactants to the catalyst surface and products away from it is most important. It is widely recognized that for a given unsaturated system the course of the hydrogenation reaction is predominantly governed by the availability of hydrogen on the catalyst surface. Higher ratios of adsorbed unsaturated groups to adsorbed hydrogen lead to higher selectivity and stronger isomerization. In commercial batch hydrogenators general rules for increasing selectivity and isomerization are to change operating conditions, causing a low concentration of hydrogen at the catalyst surface, by increasing the temperature and catalyst concentration (and activity) or decreasing the hydrogen pressure and the degree of agitation as the transfer of hydrogen from the gas phase to the liquid phase is a controlling step.

During hydrogenation of fatty oils not only the hydrogen transport but also the transport of the triglyceride molecules in the body of catalyst should be considered. In catalysts generally used considerable number of rather narrow pores are present [18], covering the range of widths between 10 and several hundreds of Angström. Diffusion of reactants into and products out of the pores may be controlling transfer-step, not effected by the type of agitation. Transport of glycerides is more difficult than that of hydrogen. Coenen *et al.* [18, 20] distinguish three pore sizes for nickel catalyst: 1) those with a diameter less than 20 Å, which are inactive in terms of hydrogenation; 2) those with a diameter 100 Å or more, considered as wide or big pores, which allow easy diffusion since there is good correlation between catalyst activity and surface area; 3) those with a diameter in between.

Selectivity can be correlated with the ratio of the number of wide pores to intermediate pores. The catalyst with narrow pores hydrogenates much less selectively than the one with wider pores [18, 52]. The narrow pores cause more stearic acid and less trans acids. Methyl esters of fatty acids having a molecular weight about one-third that of the triglycerides, diffuse in the pores more readily than the triglyceride molecules [20].

The catalyst activity as well as selectivity and isomerization [6, 60, 61] are also influenced by the partial poisoning of the catalyst. Relatively little information is available in the literature regarding this problem and there are still contrary conclusions. The alterations in catalyst activity are believed to be due to the selective adsorption of specific substances present in the reacting system [1, 6, 12, 21, 23-27, 45, 47-49, 51, 60, 61]. However, in our studies we have stated that there is very low or no influence of individual poisons on the selectivity and isomerization.

The catalytic hydrogenation of fatty oils is an extremely complex process. It is impossible to describe all its aspects in a short review.

I am aware that a lot of problems has been completely neglected and these subjects that I have touched could not been treated about in details.

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B. Drozdowski

MECHANIZM KATALITYCZNEGO UWODORNIENIA TŁUSZCZÓW

Streszczenie

Opisano strukturę i właściwości metali katalizujących reakcję uwodornienia olefin w układzie heterogenicznym oraz przedstawiono współczesne poglądy na przebieg chemisorpcji substratów na aktywnej powierzchni katalizatora. Omówiono teoretyczne podstawy problemu uwodornienia trójglicerydów z uwzględnieniem mechanizmu reakcji przyłączenia wodoru do nienasyconych acyli oraz reakcji izomerizacji pozycyjnej i geometrycznej wiązań etylenowych. Przedyskutowano wpływ podstawowych parametrów na kinetykę oraz na mechanizm reakcji. Na przykładzie katalizatora niklowego i miedziowego omówiono selektywność procesu oraz metody jej wyznaczania.

Б. Дроздовски

МЕХАНИЗМ ГЕТЕРОГЕННОЙ КАТАЛИТИЧЕСКОЙ ГИДРОГОНИЗАЦИИ ЖИРНЫХ МАСЕЛ

Резюме

Описываются структура и свойства металлов катализирующих реакцию гидрогенизации олефинов в гетерогенной схеме и рассматриваются современные взгляды на ход химisorбции субстратов на активной поверхности катализатора. Обсуждаются теоретические основы проблемы гидрогенизации триглицеридов с учетом механизма реакции присоединения водорода к ненасыщенным ацилам и реакции позиционной и геометрической изомеризации этиленовых связей. Обсуждается влияние основных параметров на кинетику и механизм реакции. На примере никелевого и медного катализаторов обсуждается селективность процесса и методы ее определения.