EFFECT OF INITIAL SOLVENT SLURRY INSIDE THE REACTOR FOR FISCHER-TROPSCH SYNTHESIS

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Abstract. *The effect of starting solvent on the performance of cobalt-based catalysts and potassium precipitated iron catalysts was investigated during Fischer-Tropsch (FT) synthesis using a continuously stirred tank reactor. In this study, starting solvents of four different molecular weights were tested: Polywax-3000 (MW (average molecular weight) = 3000), Polywax-2000 (MW = 2000), Polywax-500 (MW = 500) and C-30 oil (MW = 420). Conversion and selectivity (methane, C5?, and CO2) were similar for all starting solvents tested for potassium-supplied precipitated iron catalysts, with significant differences in starting solvent for the iron catalyst tested. there is. indicates no effect. However, with the cobalt catalyst, the conversion varied with the solvent, with the conversion increasing as the molecular weight of the solvent decreased. This is believed to be due to the particle size of cobalt alumina catalysts compared to the iron used. Under synthesis conditions, the iron catalyst produces a measurable fraction of 1-3 micron size particles in the lower range of the particle size distribution, while the alumina support maintains the same, larger size during synthesis. Thus, the decrease in conversion with time may be the result of solvent filling of the pores in the interior of the catalyst, which increases with increasing molecular weight of the starting solvent. Wax formation must be considered when deriving conversion and aging data for FT catalysts.*
Keywords: *Fischer–Tropsch synthesis, start-up solvents slurry reactor, molecular weight distributions, synthesis, reactor, cobalt and iron catalysts.*

REAKTOR ICHIDAGI DASLABKI SUSFINZIYALI QATLAMNI FISCHER TROPSCH SINTEZIGA TA'SIRI

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Annotatsiya. *Kobaltga asoslangan katalizatorlar va kaliy cho'kma temir katalizatorlarining ishlashiga boshlang'ich erituvchining ta'siri Fisher-Tropsch (FT) sintezi davomida doimiy aralashtiriladigan tank reaktoridan foydalangan holda o'rganildi. Ushbu tadqiqotda to'rt xil molekulyar og'irlikdagi boshlang'ich erituvchilar sinovdan o'tkazildi: Polywax-3000 (MW (o'rtacha molekulyar og'irlik) = 3000), Polywax-2000 (MW = 2000), Polywax-500 (MW = 500) va C-30 moyi (MVt = 420). Konversiya va selektivlik (metan, C5? va CO2) kaliy bilan ta'minlangan cho'kma temir katalizatorlari uchun sinovdan o'tgan barcha boshlang'ich erituvchilar uchun o'xshash edi, sinovdan o'tgan temir katalizatori uchun boshlang'ich erituvchida sezilarli farqlar mavjud, ta'siri yo'qligini ko'rsatadi. Biroq, kobalt katalizatori bilan konvertatsiya erituvchiga qarab o'zgarib turdi, erituvchining molekulyar og'irligi kamayishi bilan konversiya ortdi. Bu ishlatiladigan temirga nisbatan kobalt alyuminiy katalizatorlarining zarracha hajmiga bog'liq deb hisoblanadi. Sintez sharoitida temir katalizatori zarracha hajmi taqsimotining pastki oralig'ida 1-3 mikron o'lchamdagi zarrachalarning o'lchanadigan qismini ishlab chiqaradi, alyuminiy tayanchi esa sintez paytida bir xil, kattaroq hajmni saqlaydi. Shunday qilib, vaqt o'tishi bilan konversiyaning pasayishi katalizatorning ichki qismidagi teshiklarni erituvchi bilan to'ldirish natijasi bo'lishi mumkin, bu esa boshlang'ich erituvchining molekulyar og'irligi oshishi bilan ortadi. FT katalizatorlari uchun konvertatsiya ma'lumotlarini olishda mum hosil bo'lishini hisobga olish kerak.*

Kalit so'zlar*: Fisher-Tropsh sintezi, boshlang'ich erituvchilar atala reaktori, molekulyar og'irlik taqsimoti, sintez, reaktor, kobalt va temir katalizatorlari.*

ВЛИЯНИЕ ИСХОДНОГО РАСТВОРИТЕЛЯ ВНУТРИ РЕАКТОРА ДЛЯ СИНТЕЗА ФИШЕРА-ТРОПША

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Аннотация. *Влияние исходного растворителя на характеристики катализаторов на основе кобальта и железных катализаторов, осажденных калием, исследовали во время синтеза Фишера-Тропша (FT) с использованием резервуарного реактора с непрерывным перемешиванием. В данном исследовании были испытаны исходные растворители четырех различных молекулярных масс: Поливакс-3000 (МВ (средняя молекулярная масса) = 3000), Поливакс-2000 (МВ = 2000), Поливакс-500 (МВ = 500) и масло С-30. (МВт = 420). Конверсия и селективность (метан, С5? и CO2) были одинаковыми для всех исходных растворителей, протестированных для осажденных железных катализаторов с калием, со значительными различиями в исходном растворителе для протестированных железных катализаторов. есть. указывает на отсутствие эффекта. Однако при использовании кобальтового катализатора конверсия варьировалась в зависимости от растворителя, причем*

конверсия увеличивалась по мере уменьшения молекулярной массы растворителя. Считается, что это связано с размером частиц катализаторов на основе оксида алюминия-кобальта по сравнению с размером частиц используемого железа. В условиях синтеза железный катализатор производит измеримую фракцию частиц размером 1-3 микрона в нижнем диапазоне распределения частиц по размерам, в то время как носитель из оксида алюминия сохраняет тот же более крупный размер во время синтеза. Таким образом, снижение конверсии со временем может быть результатом заполнения растворителем пор внутри катализатора, которое увеличивается с увеличением молекулярной массы исходного растворителя. Образование парафина необходимо учитывать при получении данных о конверсии и старении катализаторов ФТ.

Ключевые слова: синтез Фишера–Тропша, пусковой суспензионный реактор с растворителями, молекулярно-массовые распределения, синтез, реактор, кобальтовые и железные катализаторы.

Introduction. Fischer-Tropsch synthesis (FTS) is an industrially established process for the production of predominantly linear hydrocarbons with a wide chain length distribution from synthesis gas (syngas). Considering the trend of increasing crude oil prices and stricter environmental regulations regarding sulfur content in recent years, FTS is an alternative route for the production of petrochemical substitutes and high-quality fuels. This interest is mainly due to the possibility of monetization of abundant coal reserves, remote natural gas fields and renewable biomass resources independent of oil supply. FT-derived (or synthetic) diesel fuel exhibits a much higher cetane content (typically above 70) than that obtained from crude oil at conventional refineries; fur fusion, they are almost free of environmentally harmful sulfur and have very little aromatic content. Following the successful large-scale deployment of FTS technologies by Sasol and Shell, the energy industry is considering the use of this technology as an alternative to compensate for dwindling crude oil resources. In order to improve the economics of the FTS process for industrial applications, great efforts have been made to develop more efficient catalysts and explore new technologies. Depending on the H_2/CO ratio of the feed, FTS is not easily performed with cobalt- or iron-based catalysts. Due to the low feed ratio of H_2/CO, iron catalysts are often preferred for conversion of coal-derived, CO-rich syngas because water gas shift (WGS) activity adjusts the H_2/CO ratio upwards. stoichiom etry required to perform FTS. Thus, CO_2 is rejected in the process. To limit net CO_2 emissions, biomass can be mixed with coal for gasification. Currently, the coal-based, slurry-phase FTS process uses precipitated iron catalysts supported by potassium and copper (e.g). On the other hand, cobalt catalysts are commonly used in gas-to-liquid (GTL) technology because they have a low intrinsic WGS activity suitable for converting H_2-rich, natural gas-derived syngas. Fixed and fluidized beds as well as slag bubble columns are reactor configurations used for commercial FTS. The main advantages of the hopping reaction over other technologies are that (a) the heat is easier to remove and therefore the overall

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exothermic reaction is better controlled; (b) cheaper and simpler to build, operate and maintain; and (c) the specific volume is smaller (for similar production). On the other hand, this type of reactor has a low single conversion; In addition, the catalyst erodes over time and can age faster [9]. Reactor performance may vary due to differences in fluid dynamics and dimensional characteristics of each system. In fact, it is common practice to load the slurry reactor with the start-up solvent and then suspend the fresh catalyst before starting the reactor. Starting solvents with different molecular weights can have measurably different mass and heat transfer effects during FT catalysis. A lighter solvent may have better mass transport than a heavier one, but it may leave the slurry reactor faster in the vapor phase. When high space velocities and relatively high temperatures are used for in situ reduction and FT reaction, if a light solvent is used, the slurry level in the CSTR may drop rapidly, leading to a rapid drop in conversion. Anderson et al.reviewed a qualitative picture of liquid filling of pores during F-T synthesis. The effect of the initial wax medium on a low-alpha iron FTS catalyst was studied by Gormley et al. They found that the initial wax med ium had little effect on the initial conversion rate but a large effect on the quench rate. Satterfield and Stenger the effect of liquid composition on the efficiency of slurry FT synthesis of molten magnetite catalyst. They observed that the catalyst worked well in phenanthrene, n-octacosane, and triphenylmethane, while it was quickly deactivated in Fomblin YR. The purpose of this study is to determine the effect of starting solvent on the conversion rate, selectivity and aging properties of

cobalt catalysts and potassium iron catalysts during FT synthesis using a slurry reactor.

Experimental. Precipitated iron catalysts were prepared using iron nitrate solution obtained by dissolving iron(III) nitrate nonahy drate (1.17 M) in deionized water and then adding tetraethylor tosilicate to provide the desired Fe:Si ratio. The mixture is stirred vigorously until the tetraethyl orthosilicate is hydrolyzed. A stream of tetraethylorthosilicate and ferric nitrate mixture was added to the CSTR precipitator along with a stream of ammonium hydroxide (15.6 M), which was added at a rate to maintain pH 9.0. The slurry from the CSTR was filtered using a vacuum drum filter and the solids were washed twice with deionized water. The final filter cake was dried in an oven at 110 C with flowing air for 24 h. For this study, the Fe:Si catalyst base powder was impregnated with the required amount of aqueous KNO_3 solution to produce the desired composition of Fe:Si:K = 100:4.6:1.25 (atomic ratios). After impregnation, the cat analyte was dried overnight at 110 C with good mixing and calcined at 350 C for 4 h in air flow. Condea Vista Catalox (high purity c-alumina, 150 m2/g) was used as a support for the cobalt catalyst. The catalyst was prepared by slurry impregnation and cobalt nitrate was used as a precursor. Following Sasol's patent, the solution volume to alumina weight ratio used in this method was 1:1, and the solution volume was approximately 2.5 times the pore volume of the catalyst. Two impregnation steps were used, each to load 12.5 wt% Co. Between each step, the catalyst was dried in a rotary evaporator at 80 C under vacuum and the temperature was gradually raised to 100 C. After the second impregnation/drying step, the catalyst was

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calcined at 350 C under air flow. Promoter added. The precursor used for the initiation of wet impregnation and addition of precious metals was tetraammineplatinum(II) nitrate. After the addition of Pt, the sample was dried and calcined again under the same conditions used previously. In this study, potassium iron catalysts were pretreated at CO 270 C for 24 h in a CSTR suspended in the original solvent. Carbon monoxide activation was performed at 1.3 MPa. In a typical test for cobalt catalysts, the catalyst (*12 g) was ground and sieved to obtain a 20-100 micron fraction before being loaded into a fixed-bed reactor for ex-situ reduction at 350 C and atmospheric pressure for 10 h. a H_2/He gas mixture with a molar ratio of 1:3. The contents of the hydraulic flow reactor are separated from the atmosphere by valves; the fixed-bed reactor was then connected to the CSTR through a pipe and valve purged with inert gas. The fixed-bed reactor was overpressurized with an inert gas, the valve was opened, and the catalyst was transferred to a CSTR with 310 g of liquid-phase starting solvent. The catalyst was refluxed in flowing H2 at 230 C for another 24 h. After adjusting the temperature to 40 C below the reaction temperature, the pressure was increased to the reaction pressure using a stream of H2 used during synthesis. A flow of CO was then started and the temperature was slowly increased to the reaction temperature for 2 h. FTS conditions were as follows: 220 C, 2 MPa, H_2/CO = 2.0 and SV = 3 NL/h/g-cat. FTS experiments were conducted using a 1 L CSTR equipped with a magnetically driven stirrer with a turbine impeller, a gas inlet line, and a steam outlet line with a stainless steel (SS) filter (2 lm) located outside the reactor. A tube fitted with a SS filter (0.5 lm orifice) extending

below the reactor liquid level was used to draw off the reactor wax (ie, the reactant which was solid at room temperature), thereby maintaining a relatively constant liquid level in the reactor. . Separate mass flow controllers were used to control the hydrogen and carbon monoxide flow rates. Carbon monoxide was passed through a crucible of lead oxide on alumina to remove traces of iron carbonyl. The gases are premixed in an equalization vessel and fed to the CSTR under a stirrer operating at 750 rpm. The reactor temperature was kept constant (±1 C) using a temperature controller. After the iron catalyst was activated by carburization, syngas was introduced at 6 NL/h g-Fe SV. The reaction conditions were 270 C, 1.3 MPa, H_2 / CO = 0.7 and a stirrer speed of 750 rpm. Gas, water, oil, light wax and heavy wax samples were collected and analyzed daily. Heavy wax samples were collected in a 200 C hot trap connected to a filter. The vapor phase in the region above the reactor melt was continuously transferred to hot (100 C) and then cold (0 C) traps located outside the reactor. A light wax and water mixture was collected daily from the warm trap and a water sample from the oil and cold trap.

Results and Discussion. Polywaxes (e.g., polyethylene) are saturated homopolymers of ethylene that exhibit a high degree of linearity and crystallinity. The physical properties of various start-up solvents are summarized in Table 1. These have narrow molecular weight distributions with a typical polydispersity (Mw/Mn) of 1.08. Product densities at 25 C range from a low of 0.80 g/cc for C-30 oil to a high of 0.98 g/cc for Polywax 3000. With increasing molecular weight of the start-up solvent, the average carbon number, viscosity, and

melting point increase. For FT applications, they exhibit sharp melting points, fast recrystallization, low melt viscosities, excellent heat stability and good resistance to chemical attack.

stable conversion. After reaching steady-state, essentially the same conversion level was exhibited with all of the start-up solvents tested. No significant effect on activity was observed by using different

Table 1

Physical properties of various start-up solvents.

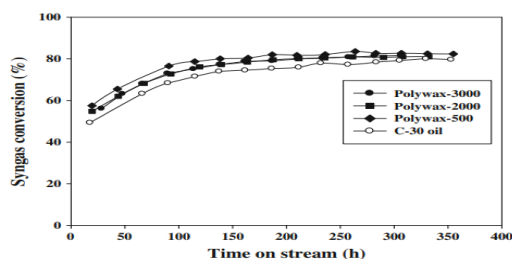| Start-up solvent	Molecular weight	Average carbon number	Density, g/cc at 25 °C	Viscosity at 149 °C	Melting point (°C)
C-30 oil	437	30	0.80	1.9	−68
Polywax-500	500	35	0.93	5	88
Polywax-2000	2,000	140	0.97	50	126
Polywax-3000	3,000	210	0.98	130	129

Fig. 1 Effect of start-up solvent media on syngas conversion for iron-based catalyst. (Reaction conditions: T = 270 C, P = 13 atm, H2/CO = 0.7, SV = 6 SL/gFe/h)

Fig. 2 Effect of start-up solvent media on syngas conversion for cobalt based catalyst. (Reaction conditions: T = 220 C; P = 20 atm; SV-3 sl/gcatalyst; H2/CO = 2)

The polywaxes used in this work are solid at room temperature; however, C-30 is a viscous liquid at room temperature. The choice of start-up solvent during FT synthesis over both cobalt catalyst and potassium-promoted precipitated Fe catalyst was studied. To maintain experimental control, similar activation and reaction conditions were maintained while the start-up solvents were varied. The effect of starting up solvent on syngas conversion for K-promoted iron catalyst is shown in Fig. 1. At similar conditions (temperature, pressure and GHSV) but varying the start-up solvent, similar induction times were observed, during which the catalyst showed increasing conversion from a low initial point to a
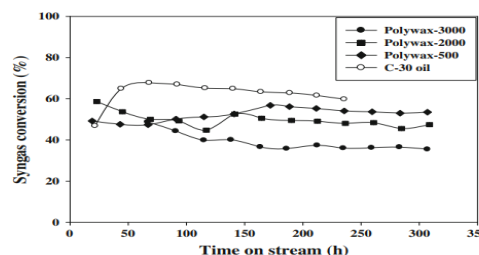
startup solvents in a slurry reactor for iron catalysts. Gormley et al. [12] observed for a low-alpha iron catalyst that as the molecular weight of the initial wax increased the catalyst deactivated more rapidly; however, this did not occur in the present study. However, for the cobalt catalyst the syngas conversion was found to vary with solvent (Fig. 2). This is likely due to the particle size of the iron catalysts being small relative to the cobalt-alumina particles used. An unsupported iron catalyst is easily broken up into fine particles in a CSTR. Supported cobalt catalysts have larger particle sizes and better attrition resistance than iron catalysts, so that a suitably calcined alumina catalyst does not attrit as fast during extended use in

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a CSTR. Figure 2 shows the syngas conversion of the cobalt cat alyst with different start-up solvents. Similar initial conversion was observed with all of the startup solvents. Using C-30 oil the syngas conversion initially increased during the next two days. With Polywax-3000 solvent, although high initial conversion was observed, it decreased in 100 h and then reached a steady-state level. With Polywax-2000 slightly higher conversion was obtained relative to Polywax-3000. Similarly, higher conversion and lower deactivation rate were found with Polywax-500 and C-30 oil solvents than with the higher molecular weight solvents.

CO conversion was observed to increase according to the following trend in the startup solvent average molecular weight: Polywax 3000 \ Polywax-2000\Polywax-500 \ C-30 oil. That is, conversion increases as the molecular weight of the solvent decreases in the range tested. Chao and Lin have reported differences in solubilities, mass transfer coefficients and diffusion coefficients in waxes of varying molecular weight. They concluded that the solubility of the syngas decreases as the molecular weight of the wax increases. If solubility or mass transport defines the conversion, it should happen for both the iron and cobalt catalysts. In this study, for the iron catalyst there is no effect of start-up solvent on conversion, whereas for the cobalt catalyst conversion was found to increase with decreasing molecular weight of the start-up solvent. An explanation based on solubility differences may be ruled out. Table 2 shows the effect of start-up solvent on the selectivity of an iron catalyst. In FT synthesis it is known that the conversion level influences the

selectivity, in part due to increasing partial pressure of water and decreasing partial pressure of reactants. It is therefore important to compare the catalysts at a similar CO level. Experiments were run with the iron catalyst at a constant conversion of CO of ca. 70%. For all start-up solvents the selectivity was found to be essentially the same: methane selectivity was *8 %, C5? selectivity was *65 % and the CO_2 selectivity was *47 %. Chain growth probability was also found to be similar for all the solvents used (a * 0.77). For the cobalt catalysts, the selectivities were also found to be identical at a similar CO conversion level (results, for the sake of brevity, are not presented). The rates of carbon monoxide consumption at steady state conversion level for cobalt and iron based catalysts as a function of start-up solvent molecular weight are shown in Fig. 3. The rate of CO consumption was found to be constant for all start-up solvents with the iron catalyst, showing that there is no effect of start-up solvent. For the cobalt catalyst, the rate of CO conversion was found to decrease with increasing molecular weight of the start-up solvent. This effect might be due to pore filling of the interior of the catalyst with increasing start-up solvent molecular weight, so that rate also follows a similar trend. Thus, the conversion is almost inversely related to the molecular weight of the solvent (Fig. 3). An experiment was carried out to further confirm that higher molecular weight start-up solvents result in lower conversion than lower molecular weight solvents using a cobalt catalyst. Initially, the reaction was started with C30 oil as the start-up solvent and similar reaction conditions; under these conditions syngas conversion was found to be *52%

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and steady state conversion was achieved around 60 h on stream (Fig. 4). After 110 h of time on stream 200 mL of hot Polywax-3000 were introduced to the reactor within a 2 h time frame. Prior to introducing the

solvent, interior pore filling is more intense than with lower molecular weight solvents for cobalt catalysts. Higher molecular weight sol vents build up on the surface, and in the catalyst pores, so that these waxes

Table 2.

Effect of start-up solvent media on activity and selectivity for Fe based catalyst

Startup solvent	Chain growth probability (α)	Selectivity (%)		
		$CH_{4\ (CO2\ free)}$	$C_{5+\ (CO2\ free)}$	CO_2
C-30 oil	0.771	8.3	65.1	47.6
Polywax-500	0.767	8.2	63.7	47.7
Polywax-2000	0.765	7.9	64.7	46.4
Polywax-3000	0.775	7.9	64.8	47.7
Reaction conditions: $T = 270\ °C$; $P = 13$ atm; SV-6; $H_2/CO = 0.7$				

Fig. 3

Effect of start-up solvent molecular weight on CO rate for iron and cobalt catalysts

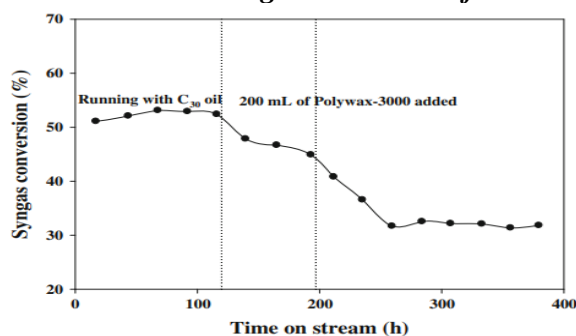**Fig. 4** Effect on syngas conversion with the addition of higher molecular weight solvent. (Reaction conditions: T = 220 C; P = 20 atm; SV-5 sl/gcatalyst; H2/CO = 2

Polywax-3000 solvent, reactor wax was withdrawn, thereby maintaining a relatively constant liquid level in the reactor. The external addition of higher molecular weight solvent caused a significant decrease in syngas conversion, as shown in Fig. 4. At around 200 h of time on stream, once again the reactor was drained and 200 mL of hot Polywax-3000 were reintroduced. Syngas conversion further declined to 32%, reaching a steady state level at approximately 250 h of time on stream. With the addition of higher molecular weight

inhibit both adsorption and diffusion rates.

They reported that olefin termination is reversible and that the diffusivity of olefins decreases rapidly with increasing carbon number; thus, the higher olefins should have longer residence times and higher fugacities within the pores of catalyst pellets.

Conclusions. The effect of the starting solvent on the conversion of cobalt cats and potassium iron catalyst was investigated using a CSTR. By varying the initial solvent molecular weight, the conversion was found to be independent of the solvent for the iron

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catalyst, while it decreased with increasing solvent molecular weight for the cobalt catalyst. Basically, the constant conversion of the small-particle iron catalyst suggests that diffusion of reactants or products in the solvent is not responsible for the differences observed for the large-particle cobalt catalyst. In contrast, the effect observed with the cobalt catalyst is probably due to the filling of the pores in the interior of the catalyst particle, and the degree of pore filling increases with increasing solvent molecular weight. A high molecular weight solvent accumulates on the surface and inside the pores of the catalyst; these waxes inhibit adsorption and slow the rate of diffusion. At a constant CO conversion level, selectivities were found to be similar for all tested starting solvents for iron and cobalt catalysts. The effect of wax accumulation within the catalyst particles should be determined by comparing the catalyst activity between different catalysts.

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