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ORGANIC DEPOSIT REMEDIATION USING ENVIRONMENTALLY BENIGN SOLVENTS: A REVIEW

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ABSTRACT

Asphaltene and paraffin wax remains the predominant organic deposit causing depositional problems in the production system. Different methods employed to remedy the problem most times has yielded different results. Chemical approach has proven quite effective due to its near hundred percent resolution of the problem. It is unfortunate that the frequently employed chemical solvents are not environmentally friendly. To this end, green solvent derived from natural and renewable sources are been utilized as an alternative to the conventional solvents for organic deposit remediation. This work critically reviews green solvents: terpene, methyl ester, ethyl lactate and cardanol. These solvents have inherent properties that make them good potential for asphaltene and paraffin wax deposit remediation. A further combination of these green solvent with environmentally friendly surfactants greatly improves their performance to remedy organic deposits. Despite the natural abundance of raw materials for the production of these green solvents, its commercialization and utilization is not wide spread.

Keywords: organic deposit, green solvent, asphaltene, paraffin wax, remediation.

INTRODUCTION

Chemical solvents are found to be effective in dissolving asphaltene and other organic deposits. These solvents include chlorinated solvents, petroleum product distillates and aromatic solvents and have been reported to be successful in treating asphaltene deposits (Newberry and Barker, 1985; Moricca and Trabucchi; 1996; Frost *et al*. 2008). In contrast, despite their solvating power for organic deposits, these solvents poses health, safety and environment risks due to their physico-chemical properties. One important property of these solvents is their peculiar low flash points which causes concern regarding their handling, transportation and storage because they are termed as highly flammable substances (Lalit and Achala, 2012). Other concerns regarding the use of these solvents as oilfield remediation solvents are their acute toxicity and environmental contamination. As a result, these solvents are on the receiving end of stricter governmental regulations, environmentalist agitations, and safety compliance issues. Therefore, an innovative green solvent is required to overcome these hurdles posed by chlorinated, aromatic and petroleum distillate solvents. OSPARCOM (Oslo Paris Commission) an environmental regulation for the European community stipulates certain guidelines for oilfield chemicals to be used in the North Sea offshore region (Kelland, 2009; Jack Dunlop, 2003). These guidelines include:

- Toxicity (EC50/LC50) >10mg/L
- Biodegradation 28days > 60% or 5days > 20%
- Bioaccumulation $\log P_{ow} \geq 3$

Organic deposits in the petroleum industry are problematic and have caused tremendous loss of crude oil production. Organic deposits include: asphaltene, paraffin wax, resins, diamondoids, organometallic, mercaptans and their combinations (Mansoori, 2010). Most pervasive with wide occurrence is asphaltene and paraffin wax as they are reported to deposit in the near wellbore region, surface and sub-surface facilities and refinery facilities (Leontaritis, 1989; Jamaluddin *et al*. 1996). These organic deposits remain in the state of thermodynamic equilibrium in the crude oil until an external factor causes its destabilization leading to the precipitation and deposition of the organic deposit (Leontaritis, 1989). These deposits are not only deemed as troublesome to control but their occurrence causes loss of production which can invariably affect the economics of the project.

Asphaltene is regarded as the heaviest portion of the crude oil, polar with high molecular weight. Generally, asphaltene are regarded to be colloidal suspended particles in the crude oil and then peptized by resins and maltenes forming a protective sheath around the asphaltene which keeps asphaltene stable in the crude oil (Leontaritis, 1989; Okafor *et al*. 2013). The destabilization of the resins and maltenes causes them to de-peptize from the crude oil forming small micro-particles which aggregates to form a visible solid (Leotaritis *et al*. 1994; Yen, 1994), drops out of solution and deposit on charged surfaces sand, clay and metal surfaces. The primary factors causing asphaltene precipitation in crude oil is changes in temperature, pressure and composition of which changes in pressure and composition are the active causative factors (Trbovich and King, 1991). Asphaltene are usually defined as a solubility class, defined to be insoluble in n-pentane or n-heptane but soluble in benzene, toluene and xylene.

Petroleum waxes are complex mixtures of n-alkanes, i-alkanes, and cylco-alkanes with carbon numbers ranging from 18 to 65. Waxes composed primarily of normal alkanes crystallize in large flat plates (macro-crystalline structures) and are referred to as paraffin waxes. Waxes composed primarily of cyclo-alkanes and i-alkanes crystallize as a small needle structures and are referred to as micro-crystalline structures. Solid paraffin

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wax formation consists of two stages: nucleation and crystal growth. As temperature of a crude oil falls to the wax appearance temperature (WAT), the wax molecules form clusters (Kelland, 2009). Wax molecules continue to attach and detach from these clusters until they reach a critical size and become stable. These clusters are called nuclei and the process of cluster formation is called nucleation. Once the nuclei are formed and the temperature remains below WAT, the crystal growth process occurs as further molecules are laid down in laminar or plate-like structure. Temperature, pressure and composition play an important role in wax formation of which temperature is predominant.

Tremendous effort has been directed to confect asphaltene dissolving and dispersing solvent devoid of known health, safety and environment risks. Lightford *et al*. (2006) designed a novel solvent-water emulsion with high flash point and low toxicity. The composition of the solvent water emulsion includes high flash point heavy aromatic naptha and alkyl poly glucoside as co-solvent all emulsified in water. Laboratory testing results shows that solvent/water emulsion can effectively dissolve asphaltene; subsequently the solvent-water emulsion was deployed to remedy asphaltene deposition problem in southern Italy oilfield. Frost *et al*. (2008) utilized a high flash point and low toxicity emulsified solvent system (ESS) evaluated its performance against xylene. The evaluation was conducted on asphaltene collected from different part of the world. Lalit and Achala (2012) equally confirmed the effectiveness of ESS as an asphaltene deposit removal. In their formulation, an aqueous acid hydrochloric acid was introduced into the mix to dissolve the inorganic deposit present in the asphaltene.

Remediation of organic deposit using chemical solvent has proven to be the most effective and efficient method to dissolve and remove asphaltene. These chemicals particularly the aromatic and chlorinated solvent has found great application in the oil and gas industry. The downside of this chemicals is their inherent environmental unfriendliness, aromatic solvent are known to possess low flash points which makes handling, transportation and storage difficult. Likewise, chlorinated solvents are toxic, non-biodegradable and owing to this demerit they have been banned in some countries. James Curtis (2003) in South and Central America, aromatic solvent are used for the production of illegal drugs as a result this solvents faces strict government regulation. Issues regarding to flammability, acute toxicity and environmental contamination is a major concern and has made the utilization of this chemicals less attractive (Samuelson, 1992; Irwin *et al*, 1997).

To this end, operators and industry researchers has confected green solvent to match the performance of the conventional aromatic and chlorinated solvent used to dissolve organic deposits. Green solvents are environmentally benign solvent derived from natural and renewable resources and totally devoid of the restrictions encountered with aromatic and chlorinated solvents. These green solvent has found application in the cleaning and pharmaceutical industry either as feedstock or co-solvent (Farid Chemat, 2012). Because of the nature of these solvents they have found application in the oil and gas industry as organic deposit dissolver and can be a direct replacement for the conventional solvents (James Curtis, 2003; Sandra Berry, 2007).

This review work highlights the successes recorded utilizing green solvents for organic deposit remediation and challenges for full scale green solvent deployment for field application.

TERPENE

Terpene are composed of repeating five-carbon isoprene units, grouped as unsaturated aliphatic cyclic hydrocarbon abundant in renewable plant resources such as orange peels (d-limonene), oleoresins from pine plant (alpha and beta pinene) while others includes turpentine, citronella, carotene and many more. Terpene has low toxicity, less flammable, rapidly biodegradable and has high solvency for organic deposit comparable to aromatic solvents. Terpene can be blended with co-solvents or an environmentally friendly surfactant to improve their performance.

Phil Rae *et al.* (2001) xylene and toluene are the most used aromatic solvents to remove asphaltene and paraffin from well tubular and near wellbore region of the reservoir formation. A more environmentally friendly terpene can show equal effectiveness in dissolving asphaltene and paraffin when used alone or in combination with other solvents and surfactants. Terpene has great solvency and if combined with a surfactant package, water soluble solution can be confected which is capable of synergistically dissolving organic deposit and reverting the formation wettability to a favorable water-wet condition. James Curtis (2003) confected terpene solvent with environmentally favorable surfactant and was used to dissolve asphaltene obtained from Boscan field and Punta de Mata field in western and eastern Venezuela respectively. The solubility of asphaltene in the terpene solvent and other chemical blend were in the range of 90-98% solubility. Paraffin obtained from Albacora field pipeline in Brazil was dissolved using the terpene solvent and recorded 70-75% solubility. Further experiment was conducted with the terpene solvent using Berea sandstone core plug with crude oil high in asphaltene and paraffin content. For the asphaltene experiment, the core plug was damaged and the permeability was reduced by 88% of the original core permeability, after the injection of the terpene solvent permeability was restored to 100% indicating a successful cleaning. Paraffin experiment followed a likewise manner, the core was damaged by paraffin deposit and reduced the original permeability by 48% and subsequent terpene solvent injection restored permeability by 97%. The confected terpene solvent finds other applications in the areas of mud removal, sand control, hydraulic fracturing and cementing application. Acunto John (2008) patent claim reported a combination of terpene (d-limonene) with commercial hydrocarbon

ARPN Journal of Engineering and Applied Sciences

solvent LPA-210 to dissolve wax and asphaltene. At an alternate %weight of d-limonene and LPA-210 for both organic deposits, the synergy was able to dissolve 84-99% of wax and asphaltene. Kelvin Fisher (2013) admonishes the novel bio based terpene derived from citrus and pine plant. Comparatively, terpene possess good solvency, biodegradable, less toxic and less flammable. In a dissolution experiment, the researcher reported a successful dissolution of asphaltene and paraffin wax using terpene solution.

A critical look at the results obtained by researchers utilizing terpene for organic deposit remediation is shown in Figure-1 below. Evidently, from the reported solubility of organic deposit in terpene, terpene has good solvency for asphaltene as its solvency is greater than 90% in each case as reported by the researchers. Also, in likewise manner terpene is good solvent for paraffin wax as (Kelvin Fisher, 2013) reported a 100% paraffin wax dissolution at a prolonged soak time with the paraffin wax.

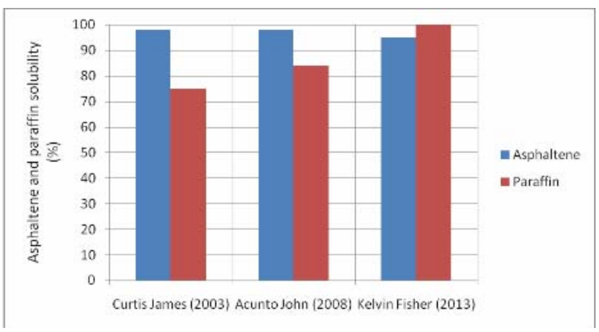

Figure-1. Comparison of organic deposit dissolution with terpene.

METHYL ESTER

Methyl ester is less toxic, biodegradable with high flash point. Methyl ester is derived from the trans-esterification of vegetable oil commonly referred to as bio-diesel because it functions in a likewise manner as the petroleum based diesel in automotive engine. Methyl ester production from vegetable oil is necessitated by the presence of methanol and catalyzed by anhydrous sodium hydroxide to yield methyl ester and glycerol as by-products (Vyas Amish *et al*. 2010; Keera *et al*. 2010). Methyl ester is polar with methyl functional group contains aliphatic tail and can participate in hydrogen bonding. Methyl ester usually contain even number of carbon atoms usually 12-24, they have long unbranched hydrocarbon chain and contain saturated and unsaturated fatty acids (John Lehman, 2009). Methyl ester of C16 and C18 are particularly of interest, though preferably carbon 18 series (Choo Yuen May *et al*, 2002). Becker (1997) proposes an asphaltene treatment with a solvent with fatty character and a hetero-atomic parent molecule.

John Willard and John Brorby (1967) in a patent claim presented the utilization of vegetable oil, alkali earth metal silicate and metal hydroxide to clean paraffin, asphaltic and waxy material from flowlines and surface facilities. The inventors injected vegetable oil into oil well and thereafter injected alkali metal hydroxide preferably sodium or potassium hydroxide into the well and finally flushing the well with water. Another variation of the invention is forming the solvent in-situ. Theoretically, the process is known as saponification when the alkali metal hydroxide contact the vegetable oil it forms a product with high dissolving and dispersing power. The inventors introduced lecithin into the mix which synergizes with the final product to enhance the dissolving potential. Alkali metal silicate preferably sodium silicate was added to the mix as a wetting agent which prevents the re-deposition of paraffin or asphaltene after cleaning. The patent claimed success in field application with the solvent as it was used to effectively clean oil wells.

Luiz Carlos Rocha *et al*. (2005) tested asphaltene inhibition in crude oil using commercially available vegetable oil. The vegetable oils used includes: pinewood essential oil, Brazil nut oil, grape seed oil, pepper Jamaican essential oil, sweet almond oil, andiroba oil, coconut essential oil, and sandal wood oil. They all showed good asphaltene precipitation inhibition out of which coconut essential oil and andiroba oil were excellent. The success of this experiment was attributed to the fact that all vegetable oil tested already contained rich organic acids and components of polyol family. Further testing for asphaltene solubilization, the vegetable oils showed no effect despite using the excellent performing coconut essential oil and andiroba oil. Palmytic acid, linolenic acid and caprylic acid tested for asphaltene inhibition in crude oil, showed excellent result but its performance is enhanced with increasing organic acids concentration. Further test for the organic acids solubilization of asphaltene showed that they quite solubilize asphaltene but less effective to commercially available dodecylbenzene sulfonic acid (DBSA) and renex 100.

Yeong *et al*. (2006) methyl ester has the ability to dissolve polar deposits. When methyl ester is applied in conjunction with surfactant their performance to dissolve polar solids (asphaltene) are greatly improved. This synergy not only dissolves but also keeps them suspended in solution thereby preventing re-deposition on clean surface. Joyce *et al*. (2009) investigated the possibility of using environmentally friendly cleaners to remove tar/asphalt from Army ground vehicles. Three cleaners were selected: terpene based cleaners, vegetable oil ester (methyl ester) and surfactant to complement both solvent performances. The three cleaners were tested in the laboratory to confirm their cleaning efficiency and the test was conducted alongside petroleum based diesel. The test was conducted severally to improve its repeatability and reproducibility and data gathered was analyzed using ANOVA analysis. Results showed that the bio based blends performed better than the petroleum based diesel. Jean Rouet *et al*. (2012) patent claimed utilized chlorophyll extracted from green leaf and methyl ester of oleic acid as co-solvent to disperse asphaltene precipitation. The extracted chlorophyll was thermally unstable and was treated with copper nitrate to improve the thermal stability of the solvent. The inventor reported that the combination of cupric chlorophyll and methyl ester rich in oleic acids was able to disperse equal amount of asphaltene with less volume as against higher volume for toxic and non-biodegradable commercial dispersants.

ETHYL LACTATE

Ethyl lactate is described as a green solvent, an alternative to traditional aromatic and chlorinated solvent, environmentally benign, and economically viable solvent. Produced via esterification of lactic acid and ethanol sourced from the fermentation of renewable substances mainly sugar rich materials or carbohydrates such as corn, wheat, rice, oats, sugar beets, cereals, sugar rich fruits and many more (Carla Pereira, 2011).

Santiago and Rafael (2008) described ethyl lactate to be characterized by intermolecular association by hydrogen bonding and alkyl group arranged in a polar domain across the liquid in a tail/tail order. Thermo-physical properties of ethyl lactate are described to be a dense fluid with moderate viscosity and thermally stable over a wide range of temperature and pressure. Ethyl lactate has solvency power incomparable to conventional solvents; as a result it has found application as a cleaning agent in cleaning metal surfaces, removing greases, oils, adhesives, paint stripper, graffiti remover. Table-1 shows the properties of ethyl lactate in comparison to conventional aromatic solvents

Table-1. Ethyl lactate properties comparison with aromatic solvents.

Property	Ethyl Lactate	Xylene	Toluene	N-methylpyrrolidone
Flash point, ^{o}C	47	28	6	91
KB value	> 1000	95	102	350
Biodegradable	Yes	No	No	No
Toxicity	No	Yes	Yes	Yes
Renewable sources	Yes	No	No	No

Sacco (2004) blended methyl ester derived from esterification of soybean vegetable oil and ethyl lactate derived from fermented ethanol and lactic acid obtained from corn sugar. The blend was used to clean asphalt from trucks, shovels and other equipment. It was reported that the bio based blend cleaning was effective and left no residue behind and proved better performance than petroleum diesel fuel. Sandra Berry *et al*. (2007) evaluated two bio based blends derived from agricultural sources, one from soybean while the other is derived from fermenting carbohydrate and whose combined kauri-butanol value is estimated to be 500. The bio based solvent was tested with composite materials such as nylon, rubber, PVC, silicone, and oilfield metals. Its visible attack and aftermath effects on the materials were observed. The exposure test with the bio based solvent to commonly used composite materials and metals in oilfield operations shows that the bio based solvent is compatible and react slightly with these materials. The bio based blend was tested for its ability to dissolve and disperse paraffin, asphaltene and drilling mud and the result compared to traditional industry solvents such as xylene and safer terpene based solvent. Test with bio based solvent on paraffin and asphaltene and drilling mud shows that the solvent can perform equally as the traditional industry solvent.

CARDANOL

Cardolite corporation submitted a report to the US EPA on cashew nut shell liquid (CNSL), the report contains environmental assessment of CNSL. CNSL contains an estimate of 70% anacardic acid, 18% cardol and 5% cardanol and other phenolic compounds. CNSL and its derivatives are essentially bio-degradable and readily soluble in water. The downside of it is that CNSL shows acute toxicity to fish and other marine organisms.

Moreira *et al*. (1999) evaluated the performance of cashew-nut shell liquid as a potential for asphaltene stabilization. CNSL is a dark viscous liquid which consist predominantly of phenolic compounds with long alkyl chains with 15 carbons having various degree of unsaturation. These phenolic compounds obtained from CNSL have a similar chemical structure as amphiphiles (nonyl phenol) studied as asphaltene stabilization agent (Chang and Fogler, 1993). Extracted from CNSL is cardanol by vacuum distillation and polymerization of cardanol was assayed in the laboratory to form polycardanol. Results of the evaluation showed that CNSL and cardanol are good asphaltene stabilizers and matched the performance of nonyl phenol while in contrast the assayed polycardanol showed poor performance and precipitated asphaltene. Palermo *et al*. (2013) gave more credence to cardanol as an effective asphaltene stabilizer. The researchers combined a commercial solvent ethylene-co-vinyl acetate copolymer (EVA33) with four asphaltene dispersants (DBSA, cardanol and two commercial products INH1 and INH2). By conducting a series of performance evaluation test which includes onset of asphaltene precipitation and wax deposition inhibition test (pour point depression). The combination of EVA 33 and cardanol was able to shift the onset of asphaltene precipitation and reduce the pour point of the crude under investigation at low concentration. The researchers ranked the formulation of EVA33 and cardanol the best performing combination for asphaltene and wax inhibition.

The major setbacks of green solvents are cost and available technology for production. Sourcing raw material is never a problem as the original sources or starting material are abundant in nature. Other issues regarding challenges of green solvent are purity as extracted starting material are in their impure form and the solvent of interest need to be isolated and extracted. Final formulation after undergoing the processes of extraction, purification and alteration processes (alkylation, sulphonation, polymerization and ethoxylation), solvents should still maintain their HSE characteristics. Due to this processes of production the cost of green solvent are deemed expensive which affects their commercial availability. But as a result of advancement of technology and less expensive production processes, the overall cost for the production of these benign green solvents will be significantly lowered.

CONCLUSIONS

Green solvent derived from natural resources has shown great potential as organic deposit remediation. As a result of the successes recorded the following conclusion can be drawn. The identified green solvents: terpene, methyl ester, ethyl lactate and CNSL are good asphaltene and paraffin wax dissolvers. In similar manner, green solvent has performed excellently as asphaltene inhibitors in comparison with commercial inhibitors. Comparison between this green solvents and conventional solvents has shown matched performance for asphaltene and paraffin wax dissolution. Green solvents are devoid of the peculiar problems witnessed when utilizing conventional solvents especially regarding their low flash point, acute toxicity and non-biodegradability. The major challenges faced by green solvent product are cost and available technology for production.

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